



Agilent 355 Sulfur and 255 Nitrogen Chemiluminescence Detectors

**Operation and Maintenance
Manual**



Agilent Technologies

Notices

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CAUTION

A **CAUTION** notice denotes a hazard. It calls attention to an operating procedure, practice, or the like that, if not correctly performed or adhered to, could result in damage to the product or loss of important data. Do not proceed beyond a **CAUTION** notice until the indicated conditions are fully understood and met.

WARNING

A **WARNING** notice denotes a hazard. It calls attention to an operating procedure, practice, or the like that, if not correctly performed or adhered to, could result in personal injury or death. Do not proceed beyond a **WARNING** notice until the indicated conditions are fully understood and met.

Warnings

English

WARNING



This symbol on the instrument indicates that the user should refer to the manual for operating instructions.

WARNING



Any operation requiring access to the inside of the equipment, could result in injury. To avoid potentially dangerous shock, disconnect from power supply before opening the equipment.

WARNING



For continued protection against fire hazard replace fuse with same type and rating.

WARNING



This symbol indicates that to comply with European Union Directive 2002/96/EC for waste electrical and electronic equipment (WEEE), the Analyzer should be disposed of separately from standard waste.

WARNING

This is a safety Class I product. It must be wired to a mains supply with a protective earthing ground incorporated into the power cord. Any interruption of the protective conductor, inside or outside the equipment, is likely to make the instrument dangerous. Intentional interruption is prohibited.

WARNING

If this instrument is used in a manner not specified by Agilent, the protection provided by the instrument may be impaired.

WARNING

High voltage is present in the instrument when the power cord is connected, even if the main power switch is in the standby mode. To avoid potentially dangerous shock, disconnect the power cord before removing the side panels.

WARNING

Ozone is a hazardous gas and a strong oxidant. Exposure to ozone should be minimized by using the instrument in a well-ventilated area and by venting the exhaust of the vacuum pump to a fume hood. The ozone generator should be turned off when the instrument is not in use.

WARNING

Burner temperature is extremely hot. Do not touch. Allow to cool before servicing.

WARNING

Hydrogen is an extremely flammable gas. Use appropriate care when handling. Inspect all connections with a suitable leak detector.

WARNING

Oxygen rich environments can promote combustion and even result in spontaneous combustion under conditions of high pressure and exposure to contamination. Use only oxygen rated components and ensure that components are oxygen clean prior to use with pure oxygen.

WARNING

Exceeding the gas inlet pressure of 25 psig (1.72 bar) may damage the hydrogen and oxidant sensors or burst their connective tubing.

Español

WARNING



Cualquier operación que requiera acceso al interior del equipo, puede causar una lesión. Para evitar peligros potenciales, desconectarlo de la alimentación a red antes de abrir el equipo.

WARNING



Para protección continua contra el peligro de fuego, sustituir el fusible por uno del mismo tipo y características.

WARNING



Este símbolo en el instrumento indica que el usuario debería referirse al manual para instrucciones de funcionamiento.

WARNING

Esto es un producto con clase I de seguridad. Debe conectarse a una red que disponga de tierra protectora en el cable de red. Cualquier interrupción del conductor protector, dentro o fuera del equipo, puede ser peligroso. Se prohíbe la interrupción intencionada.

WARNING

Si este instrumento se usa de una forma no especificada por Agilent, puede desactivarse la protección suministrada por el instrumento.

Français

WARNING



Chaque opération à l'intérieur de l'appareil, peut causer du préjudice. Afin d'éviter un shock qui pourrait être dangereux, déconnectez l'appareil du réseau avant de l'ouvrir.

WARNING



Afin de protéger l'appareil continuellement contre l'incendie, échangez le fusible par un fusible du même type et valeur.

WARNING



Le symbol indique que l'utilisateur doit consulter le manuel d'instructions.

WARNING

Ceci est un produit de Classe de sécurité I. L'instrument doit être branché sur l'alimentation secteur par un fil de secteur prévu d'une prise de masse. Chaque interruption du conducteur protégeant, à l'intérieur ou à l'extérieur de l'appareil peut rendre l'instrument dangereux. Interruption intentionnelle est interdite.

WARNING

Si l'instrument n'est pas utilisé suivant les instructions de Agilent, les dispositions de sécurité de l'appareil ne sont plus valables.

Deutsch

WARNING



Vor dem Öffnen des Gerätes Netzstecker ziehen!

WARNING



Für kontinuierlichen Schutz gegen Brandgefahr dürfen bei Sicherungswechsel nur Sicherungen der gleichen Stärke verwendet werden!

WARNING



Dieses Symbol auf dem Gerät weist darauf hin, dass der Anwender zuerst das entsprechende Kapitel in der Bedienungsanleitung lesen sollte.

WARNING

Dies ist ein Gerät der Sicherheitsklasse I und darf nur mit einem Netzkabel mit Schutzleiter betrieben werden. Jede Unterbrechung des Schutzleiters außerhalb oder innerhalb des Gerätes kann das Gerät elektrisch gefährlich machen. Absichtliches Unterbrechen des Schutzleiters ist ausdrücklich verboten.

WARNING

Wenn das Gerät nicht wie durch die Firma Agilent, vorgeschrieben und im Handbuch beschrieben betrieben wird, können die im Gerät eingebauten Schutzvorrichtungen beeinträchtigt werden.

Italiano

WARNING



Qualsiasi intervento debba essere effettuato sullo strumento può essere potenzialmente pericoloso a causa della corrente elettrica. Il cavo di alimentazione deve essere staccato dallo strumento prima della sua apertura.

WARNING



Per la protezione da rischi da incendio in seguito a corto circuito, sostituire i fusibili di protezione con quelli dello stesso tipo e caratteristiche.

WARNING



Il simbolo sullo strumento avverte l'utilizzatore di consultare il Manuale di Istruzioni alla sezione specifica.

WARNING

Questo strumento è conforme alle specifiche per i prodotti in Classe I - Il cavo di alimentazione dalla rete deve essere munito di "terra". Qualsiasi interruzione del cavo di terra all'interno ed all'esterno dello strumento potrebbe risultare pericolosa. Sono proibite interruzioni intenzionali.

WARNING

Se questo strumento viene utilizzato in maniera non conforme alle specifiche di Agilent, le protezioni di cui esso è dotato potrebbero essere alterate.

Dutch

WARNING



Iedere handeling binnenin het toestel kan beschadiging veroorzaken. Om iedere mogelijk gevaarlijke shock te vermijden moet de aansluiting met het net verbroken worden, vóór het openen van het toestel.

WARNING



Voor een continue bescherming tegen brandgevaar, vervang de zekering door een zekering van hetzelfde type en waarde.

WARNING



Het symbool geeft aan dat de gebruiker de instructies in de handleiding moet raadplegen.

WARNING

Dit is een produkt van veiligheidsklasse I. Het toestel moet aangesloten worden op het net via een geaard netsnoer. Bij onderbreking van de beschermende geleider, aan de binnenzijde of aan de buitenzijde van het toestel, kan gebruik het toestel gevaarlijk maken. Opzettelijke onderbreking is verboden.

WARNING

Indien het toestel niet gebruikt wordt volgens de richtlijnen van Agilent, gelden de veiligheidsvoorzieningen niet meer.

The following symbols are used on the equipment:



Caution - Refer to manual for operating instructions

Caution - Risk of electrical shock.

Caution - Hot surface.

Atención - Ver documentación pertinente.

Atención - Riesgo de sacudidas eléctricas.

Atención - Superficie caliente.

Attention - Consultez les documents d'accompagnement.

Attention - Risque de choc électrique.

Attention - Surface brûlante.

Vorsicht - Siehe beiliegende Unterlagen.

Vorsicht - Risiko eines Elektroschocks.

Vorsicht - Heisse Oberfläche.

Pericolo - Vedi documentazione allegata.

Pericolo - Pericolo di scosse elettriche.

Pericolo - Superficie rovente.

Voorzichtig - Raadpleeg de bijbehorende documentatie.

Voorzichtig - Hoge spanning, levensgevaar.

Voorzichtig - Heet oppervlak.

Contents

1 Introduction

2 System Description

Specifications	20
Technical Information — 355 SCD	20
Technical Information — 255 NCD	20
Physical Specifications	21
Theory of Operation	23
Sulfur Chemiluminescence Detector	23
Nitrogen Chemiluminescence Detector	23
Dual Plasma Controller	24
Description of Major Components	25
Dual Plasma Burner	25
Figure 1. Cross-Section of the Dual Plasma Burner for the 355 SCD	26
Figure 2. Cross-Section of the Dual Plasma Burner for the 255 NCD	27
Dual Plasma Controller	27
Ozone Generator	28
Chemiluminescence Reaction Cell and Photomultiplier Tube	28
Figure 3. 355 SCD Left Side	29
Figure 4. 355 SCD Right Side	30
Figure 5. 255 NCD Left Side View	31
Figure 6. 255 NCD Right Side View	32
Pressure Transducer	32
Vacuum Pump	32
Chemical Trap	33
Oil Coalescing Filter	33
FID Adapter (Optional)	33
NCD Reaction Cell	33
NCD Photomultiplier Tube and Cooler	34
Figure 7. Schematic for 355 SCD	35
Figure 8. Schematic for 255 NCD, in Nitrogen Mode	36
Figure 9. Schematic for 255 NCD, in Nitrosamine Mode	37

3 Installation

Overview	40
Step 1: Selecting a Location	41
Power Requirements	41
Figure 10. Drawing of the Detector with Dual Plasma Burner and Controller	42
Environmental Considerations	42
Combustion Gas Requirements	42
Step 2: Unpack and Inspect the Instrument	45
Required Installation Tools	45
Step 3: Set Up the Vacuum Pump	46
Installing the Edwards RV5 Pump Oil-Sealed Vacuum Pump	46
Figure 11. RV5 Oil-Sealed Vacuum Pump and Associated Traps (Front Side)	49
Figure 12. RV5 Oil-Sealed Vacuum Pump and Associated Traps (Back Side)	49
Figure 13. RV5 Oil- Sealed Vacuum Pump and Associated Traps (Top)	50
Figure 14. RV-5 Oil-S ealed Vacuum Pump Exhaust Line	51
Installing the Welch Dry Piston Vacuum Pump	52
Figure 15. The Welch Dry Piston Pump	54
Figure 16. Oil Drain Kit with Ballast Control	54
Step 4: Connect the Power Cord	56
Figure 17. SCD Rear Panel Diagram	56
Step 5: Install the Dry Compressed Air or O ₂ Supply	57
Step 6: Install the Signal Output Cables	58
Standard Cable Connection	58
HP 3390 Series Integrator Cable Connection	58
HP 3396 Integrator Cable Connection	58
HP 5890 GC Analog Input Board	58
Agilent 6890 GC Analog Input Board	58
Step 7: Install the Dual Plasma Burner	59
Figure 18. Dimensions of GC Liner Cut-Outs	59
Step 8: Install the Dual Plasma Controller	60
Figure 19. Dual Plasma Controller Rear Panel	60
Step 9: Install Column Connections	61
Capillary Columns	61
Figure 20. Measuring Column Insertion	61

Packed Columns and Columns with an Outside Diameter > 0.8 mm	61
Step 10: Install the Transfer Line	62
4 Front Panel Controls and Initial Startup	
Detector Front Panel Controls	64
Figure 21. Front Panel Controls	64
Power Controls	64
Display Output Controls	65
Signal Controls	65
Dual Plasma Controller Controls	66
Figure 22. Dual Plasma Controller Front Panel	66
Initial Startup	67
Detector Interface Setup	69
Initial Checkout	69
Monitoring Oxidizer and Hydrogen Flow with the Dual Plasma Controller	70

5 Operation

Start-Up Procedure	72
Detector Operation	73
Detector Stability and Response	73
Column Bleed	73
Coking	73
Hydrogen Poisoning	73
Contaminated Gases	74
Fluctuating Pressures	74
Typical Operating Conditions	75
Table 1. Typical Operating Conditions	75
Detection Limits	76
Table 2. Expected Detection Limits for Chromatographic Conditions	76
Instrument Shut-Down	77
Daily Shutdown	77
Complete Shutdown	77
Special Operating Modes	78
Using the 255 NCD in Nitrosamine Mode	78
Using the SCD in High Sensitivity Mode for Nonhydrocarbon Gaseous Samples	78

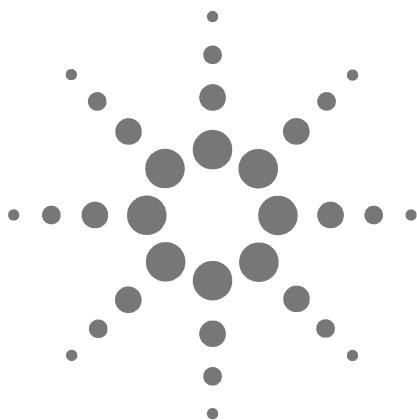
6 Maintenance

Pump Maintenance	82
Table 3. Operating Life of Components for Edwards RV5 Vacuum Pump	82
Cleaning the Detector	83
Changing the Oil Mist Filter (RV5)	84
Reaction Cell Cleaning	85
Figure 23. Reaction Cell, PMT Housing and PMT Socket	86
Figure 24. Reaction Cell	86
Flow Sensor Calibration	88
Detector Sensitivity	89
Assembling the Dual Plasma Burner for Component Replacement with the SCD	90
Figure 25. Ferrule Placement on Lower Burner Tube	90
Figure 26. Proper Ferrule Orientation to the Large Ceramic Tube	91
Figure 27. Large Ceramic Tube Properly Inserted into the Quartz Heater Assembly	91
Figure 28. Orientation of the Double Taper Ferrule	92
Figure 29. Positioning the Upper Tube in the Union Fitting	92
Figure 30. Tightening the Heater Swivel Nut	93
Figure 31. Proper Alignment of the Burner	93
Assembling the Dual Plasma Burner for Component Replacement with the NCD	95
Figure 32. Ferrule Placement on Lower Burner Tube	95
Figure 33. Proper Ferrule Orientation to the Large Quartz Tube	96
Figure 34. The Quartz Tube Properly Inserted into the Quartz Heater Assembly	96
Figure 35. Burner Assembly Detail	97
Figure 36. Burner Assembly Alignment	97
Tube Replacement for the SCD	98
Figure 37. Orientation of the Double Taper Ferrule	98
Figure 38. Positioning the Upper Tube in the Union Fitting	99
Figure 39. Proper Ferrule Orientation to the Large Ceramic Tube	99
Figure 40. Large Ceramic Tube Properly Inserted into the Quartz Heater Assembly	100
Figure 41. Tightening the Heater Swivel Nut	100
Figure 42. Proper Alignment of the Burner	101
Tube Replacement for the NCD	102
Figure 43. Proper Ferrule Orientation to the Large Quartz Tube	102
Figure 44. Large Quartz Tube Properly Inserted into the Quartz Heater Assembly	103
Figure 45. NCD Tube Replacement Detail	104
Figure 46. NCD Tube Replacement Detail	104

7 Troubleshooting

Solving Detector Problems	106
Power Problems	107
Detector Fuse	107
Vacuum Pump Fuse	107
Dual Plasma Controller Fuses	107
Table 4. Fuses for 100 V, 120 V and 230 V Versions of 355 SCD and 255 NCD	108
Figure 47. Fuse Positions on the Power Supply Board	108
Ozone Generation Problems	109
Ozone Generator	109
High Voltage Transformer	109
Plugged Restrictor Lines	110
Response Problems	111
Temperature Reading Problems	112
Diagnosing General Problems	113
Table 5. Troubleshooting Detector Issues	113
Table 6. Troubleshooting Pump Issues	115
Table 7. Troubleshooting Burner Issues	116

Index



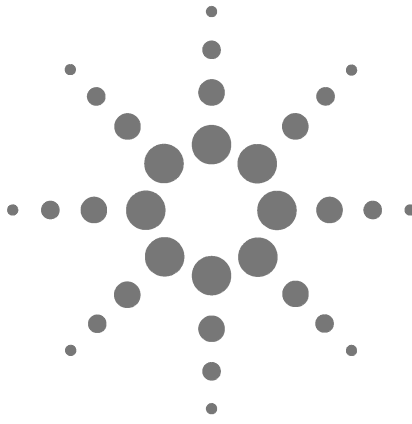
1 Introduction

This manual will guide you in the installation, operation, and troubleshooting of the Agilent 355 Sulfur Chemiluminescence Detector (SCD) and the Agilent 255 Nitrogen Chemiluminescence Detector (NCD). This manual is intended for use with the Agilent 355 SCD or 255 NCD with the Dual Plasma Burner and Controller.

This operation and service manual has some important conventions, such as the use of boxed warnings. This information is deliberately set out from the text for emphasis and should be followed to assure operator safety and proper instrumental operation.

If you are installing the 355 SCD or 255 NCD yourself, follow the installation procedures described in the following sections. If your instrument is already installed, turn to the Operation section to begin.





2 System Description

Specifications	20
Theory of Operation	23
Description of Major Components	25



Specifications

Technical Information — 355 SCD

Sensitivity*	Typical < 0.5 pg S/second (signal to noise 3.3:1)
Typical Selectivity	g S/g C > 2×10^7
Linearity	> 10^4
Precision and Stability†	<2% RSD 2 hours <5% RSD 72 hours
Ozone flow through the Post Ozone Restrictor	20-30 mL/min at 3-6 psig
Typical reaction cell pressure	4 - 8 Torr RV5 Oil Sealed Pump 6 - 12 Torr Dry Piston Pump
Typical Burner Pressure	250-400 Torr operating
Typical Burner Temperature	800 °C
Typical Air Flow Rate	65 SCCM recommended 3-10 SCCM recommended with FID adapter
Typical Hydrogen Flow Rate	40 SCCM recommended
Signal Output Ranges	0-1V, 0-10V
Typical time to reach 800 °C from room temperature	10 min typical(120 VAC, 60 Hz)
Typical safety shroud outside temperature	<65 °C at 800 °C Burner temperature typical
Recorder output	0-1 V or 0-10 V

* Burner temperature 800 °C, 80 SCCM air, and 60 SCCM hydrogen, test compound dimethyl sulfide in toluene.

† Based on thiopene in benzene at 1 ppm mass sulfur, 1 µL injection split 1:10, 30 m, 0.32 mm ID, 1 µm thick CP wax (n=10 for 2.5 hours; n=42 for 72 hours).

Subject to change without notice.

Technical Information — 255 NCD

Sensitivity	<3 pg N/second (signal to noise 3:1) in both N and nitrosamine modes
Selectivity	g N/g C > 2×10^7 in N mode (selectivity in nitrosamine mode is matrix-dependent)
Linearity	> 10^4

Repeatability*	<1.5% RSD 8 hours (~ the same in N and nitrosamine) <2% RSD 18 hours (~3% RSD in nitrosamine mode over 21 hours)
Gas flow through Ozone Generator	20-30 mL/min at 3-6 psig (inlet pressure)
Typical reaction cell pressure	4 - 8 Torr RV5 Oil Sealed Pump 6 - 12 Torr Dry Piston Pump
Typical Burner Pressure	130 - 150 torr operating
Typical Burner Temperature	900 °C
Typical Hydrogen Flow Rate	6-10 SCCM
Typical Oxygen Flow Rate	8 - 15 SCCM
Signal Output Ranges	0-1V, 0-10V, 0 - 10V

* Burner temperature 950 °C, 11 SCCM oxygen, and 6 SCCM hydrogen; 25 ppm N as nitrobenzene in toluene; 0.2 µL injection on column (HP 19095-121Z), n=7 for 3 hours; n=13 for 18 hours and n=10 n-dipropylnitrosamine in toluene at 4 µg/mL, 0.2 µL injection on column (HP 19095-121Z).

Physical Specifications

Power requirements

355 SCD Detector	115 VAC, 50/60 Hz, 1400 W 100 VAC, 50/60 Hz, 1400 W 220-240 VAC, 50/60 Hz, 650W
255 NCD Detector	115 VAC, 50/60 Hz, 1400 W 100 VAC, 50/60 Hz, 1400 W 220-240 VAC, 50/60 Hz, 650W
Dual Plasma Controller	100-120 VAC, 50/60 Hz, 200 W 220-240 VAC, 50/60 Hz, 200 W

Dimensions and weight

Detector	Height: 16.0 in (40.6 cm) Width: 9.2 in (23.4 cm) Depth: 21.8 in (55.3 cm) 355 SCD Weight: 34.0 lbs (15.0 kg) 255 NCD Weight: 37.5 lbs (17.0 kg)
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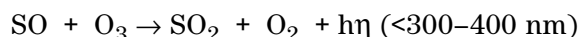
Dual Plasma Controller	Height: 5.0 in (12.7 cm) Width: 9.5 in (24.1 cm) Depth: 12.5 in (31.8 cm) Weight: 9.9 lbs (4.5 kg)
Dual Plasma Burner	Height: 12.3 in (31.2 cm) Diameter: 4.0 in (10.2 cm) Weight: 1.9 lbs (0.9 kg)
Oil Sealed Vacuum Pump (RV5)	Height: 10.3 in (26.1 cm) Width: 6.0 in (15.2 cm) Depth: 16.9 in (43.0 cm) Weight: 47.3 lbs (21.5 kg)
or	
Oil Free Dry Piston Pump	Height: 12.0 in (30 cm) Width: 9.0 in (22.9 cm) Depth: 14.0 in (35.6 cm) Weight: 29.9 lbs (13.6 kg)
Installation Category	II
Pollution Degree	2
Ambient Temperature	50 - 104 °F (10-40 °C)
Relative Humidity	Up to 95%, noncondensing
Normal Operating Environment	Intended for indoor use only
Maximum Altitude	2,000 m (6,562 ft)
Mains Supply Voltage	Fluctuation not to exceed 10% of nominal voltage

Theory of Operation

Sulfur Chemiluminescence Detector

The Agilent model 355 Sulfur Chemiluminescence Detector (SCD) is a sulfur-selective detector for gas chromatography. Operation of the SCD is based on the chemiluminescence (light-producing reaction) from the reaction of ozone with sulfur monoxide (SO) produced from combustion of the analyte:

Sulfur compound (analyte) \rightarrow SO + H₂O + other products



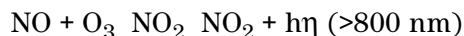
A vacuum pump pulls the combustion products into a reaction cell at low pressure, where excess ozone is added. Light produced from the subsequent reaction is optically filtered and detected with a blue-sensitive photomultiplier tube and the signal is amplified for display or output to a data system. Figure 1 is a pneumatic flow diagram that illustrates how the components of the system are integrated.

The Detector has an enclosed, dedicated (Dual Plasma) Burner designed to enhance production of the SO intermediate. This Dual Plasma Burner mounts in the detector port of the GC. A Dual Plasma Controller provides temperature control and gas-flow regulation to operate the Dual Plasma Burner.

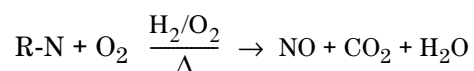
The Agilent model 355 SCD provides high sensitivity (<0.5 pg S/sec), with linear and equimolar response over four orders of magnitude (per Sulfur atom) while maintaining high selectivity over common solvents. The Agilent SCD is compatible with most commercially available gas chromatographs.

Nitrogen Chemiluminescence Detector

Operation of the Agilent model 255 Nitrogen Chemiluminescence Detector is based on the chemiluminescence or light-producing reaction of ozone with nitric oxide formed from combustion. Reacting nitric oxide with ozone results in the formation of electronically excited nitrogen dioxide. The excited nitrogen dioxide emits light, a chemiluminescence reaction, in the red and infrared region of the spectrum. The light emitted is directly proportionally to the amount of nitrogen in the sample,



The light ($h\nu$) emitted by the chemical reaction is optically filtered and detected by a photomultiplier tube. The signal from the photomultiplier tube is amplified for display or output to a data system. Organic compounds containing nitrogen react to form nitric oxide, carbon dioxide, and water.



Dual Plasma Controller

The Dual Plasma Controller provides all operational parameters of the Dual Plasma Burner except for the Detector base temperature. The Detector base temperature is controlled by circuitry in the GC. Parameters monitored or regulated by the Controller include Burner temperature, Burner temperature set-point, hydrogen and oxidant flow rates, and Burner pressure. The temperature set-point, actual pressure, oxidant and hydrogen flow rates are displayed by rotation of a 4-position switch. Power, valve operation, temperature within set-point range and fault conditions are indicated with LED illumination on the front display panel.

The Dual Plasma Controller incorporates several safety features. The safety circuitry detects faults such as power loss, vacuum loss, thermocouple failure, heater element failure, broken ceramic tube, or high temperature. When a fault is detected, the Fault LED illuminates and hydrogen and oxidant flow is stopped by normally-closed solenoid valves.

Dual Plasma Burner with the 355 SCD

The Dual Plasma Burner is based on the same chemistry and basic principles of earlier SCD Burner designs. A key difference, however, is the addition of a second “flame” or “plasma,” the lower is oxygen-rich and the upper is hydrogen-rich. The Burner consists of a tower assembly that contains an outer sheath for burn protection, a heating element, thermocouple, and combustion tubes. Conversion of sulfur containing compounds to SO occurs within the ceramic reaction chamber housed in the Burner assembly. Agilent also provides a Flame Ionization Detector (FID) option for the simultaneous collection of hydrocarbon and sulfur chromatograms for some GCs.

Dual Plasma Burner with the 255 NCD

Compounds eluted from the GC column are combusted in the Dual Plasma Burner first by an oxygen rich flame (plasma) followed by catalytic combustion on a Noble metal screen. For hydrocarbons, this two stage combustion technique results in complete conversion of the matrix to products, such as carbon dioxide and water, which do not chemiluminesce with ozone. Nitrogen atoms in a compound are converted into nitric oxide and potentially other nitrogen oxide species. In the second stage, the catalyst is used to convert other nitrogen oxide species to nitric oxide, resulting in a high efficiency of conversion.

Description of Major Components

Dual Plasma Burner

The Dual Plasma Burner consists of a tower assembly that contains an outer sheath for burn protection, a heating element, thermocouple, and combustion tubes. In the SCD, conversion of sulfur containing compounds to SO occurs within the ceramic reaction chamber housed in the Burner assembly and potentially interfering hydrocarbons are oxidized to CO₂ and H₂O, with air as the oxidant. In the NCD, oxygen is used as the oxidant.

A fitting is located on top of the Burner. The vacuum line from the Detector box is connected directly to the top of this fitting and H₂ is input to the longer side of this fitting. The air inlet is connected to the base of the Burner.

The Burner is mounted onto the GC by a model-specific mounting kit (see www.Agilent.com/chem or contact Agilent for the most current information). The GC column is inserted into the Burner using a 1/32" knurled nut and fused silica adapter ferrule.

A cross-section illustration of the Dual Plasma Burner for the 355 SCD is shown in Figure 1 and for the 255 NCD is shown in Figure 2.

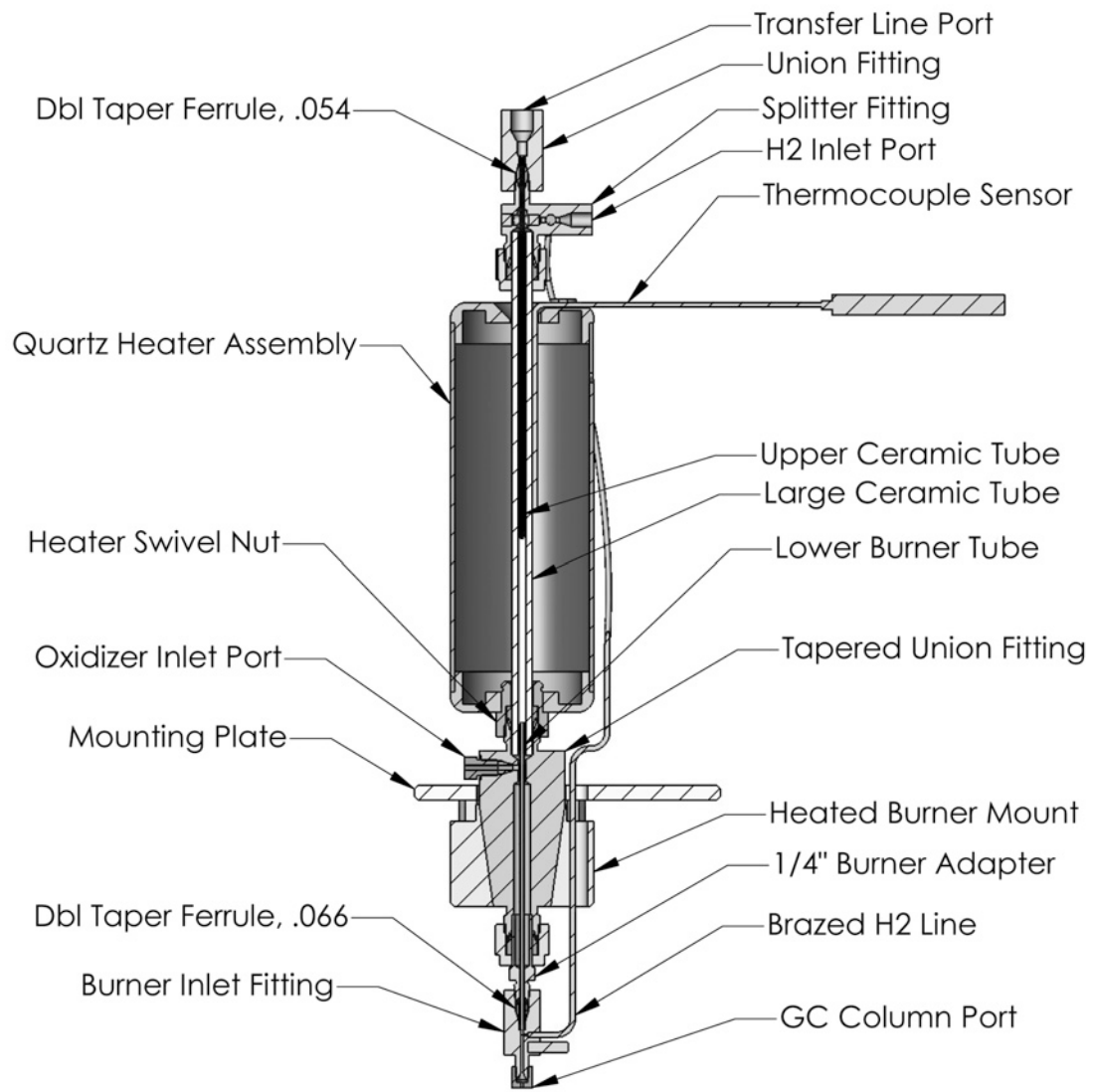


Figure 1 Cross-Section of the Dual Plasma Burner for the 355 SCD

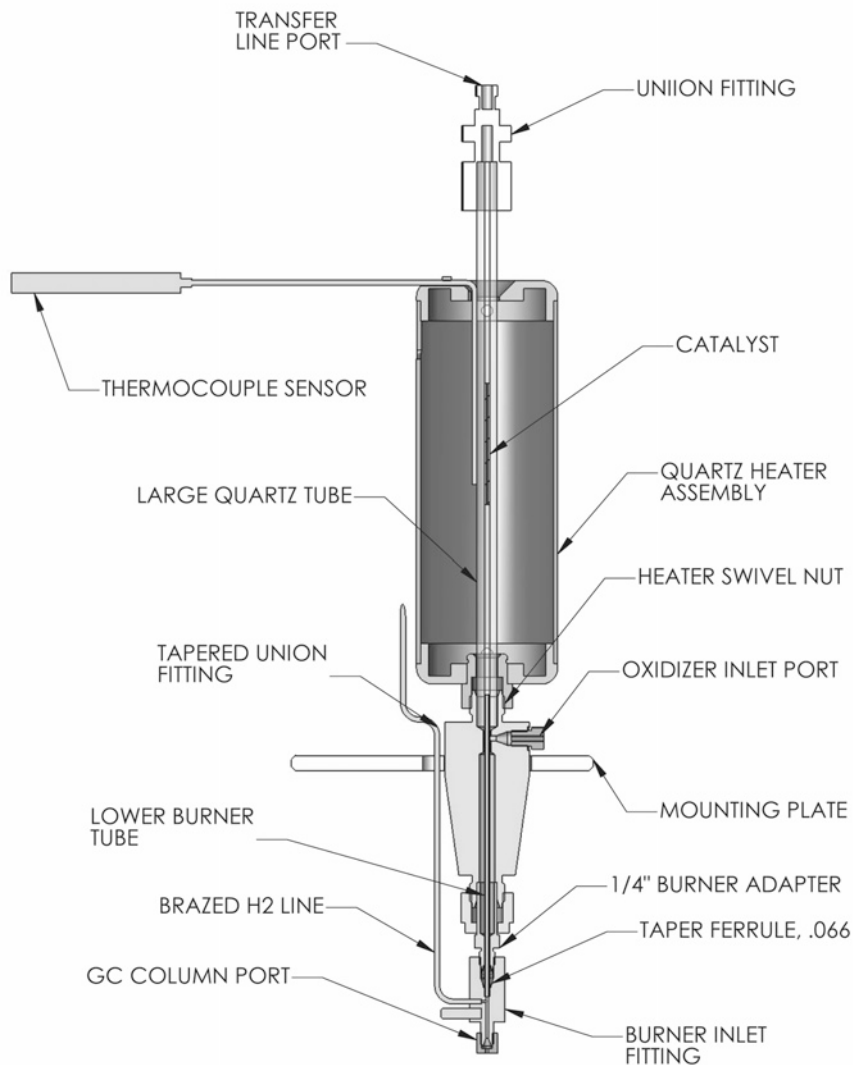


Figure 2 Cross-Section of the Dual Plasma Burner for the 255 NCD

Dual Plasma Controller

The Dual Plasma Controller powers the Dual Plasma Burner and supplies its gases. Hydrogen and oxidant should be provided at 25 psig (1.72 bar) and the Controller is plugged into an appropriate AC electrical outlet.

WARNING

Exceeding the gas inlet pressure of 25 psig (1.72 bar) may damage the hydrogen and oxidant sensors or burst their connective tubing.

The parameters monitored or regulated by the Controller include Burner

temperature, hydrogen and oxidant flow rates, and Burner pressure. The temperature, actual pressure, oxidant and hydrogen flow rates are selected for display by rotation of a 4-position control knob. Power, valve operation, temperature within set-point range and fault conditions are indicated with LED illumination on the front display panel.

The Dual Plasma Controller incorporates several safety features. The safety circuitry detects faults such as power loss, vacuum loss, thermocouple failure, heater element failure, broken ceramic tube, or high temperature. When a fault is detected, the Fault LED illuminates and hydrogen and oxidant flow is stopped by normally-closed solenoid valves.

Ozone Generator

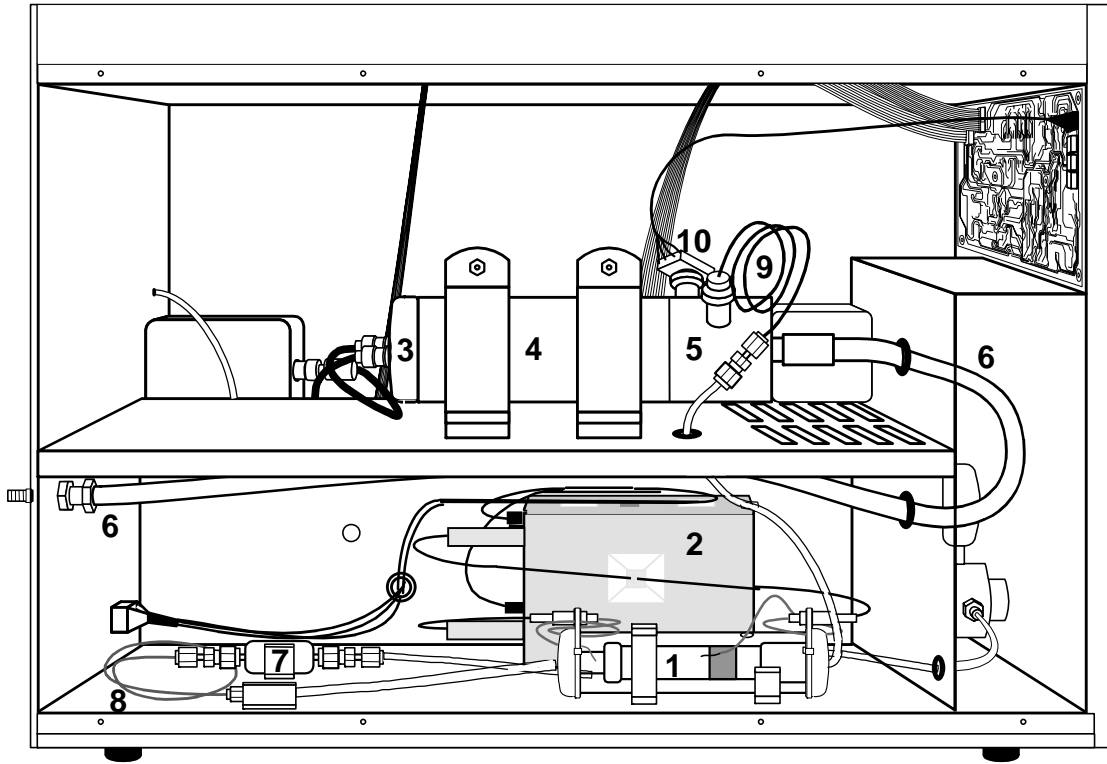
The SCD and NCD produce ozone by corona discharge using a clean, pressurized air or oxygen source. Use of oxygen should increase ozone production and, hence, Detector response. High voltage to the ozone generator is applied only when the reaction cell pressure is less than 100 torr in the SCD and less than 200 torr in the NCD. Gas flow through the ozone generator is controlled by a pressure regulator and flow restrictors.

WARNING

Ozone is a hazardous gas and a strong oxidant. Exposure to ozone should be minimized by using the instrument in a well ventilated area, changing the chemical trap regularly, and venting the exhaust of the vacuum pump. The ozone generator should be turned off when the instrument is not in use to reduce maintenance requirements.

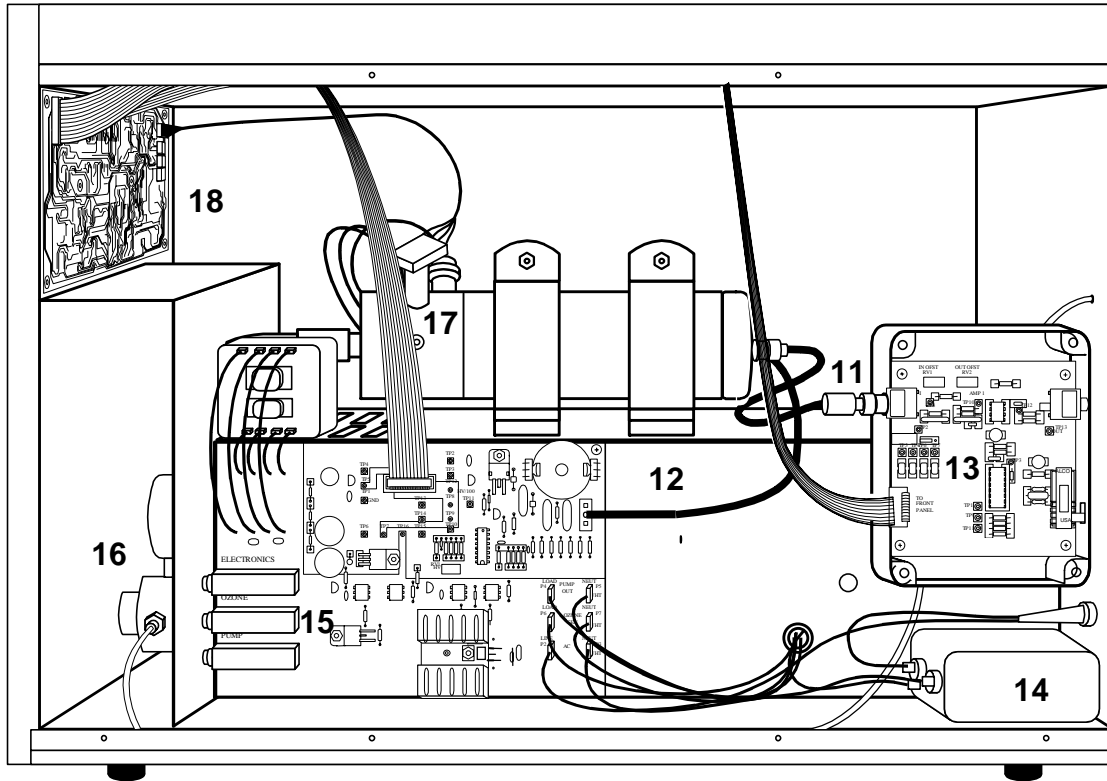
Chemiluminescence Reaction Cell and Photomultiplier Tube

Sulfur monoxide (formed in the Burner) and ozone (produced in the ozone generator located in the Detector) are mixed in the reaction cell. The cell is designed such that the reaction between SO and O₃ occurs directly in front of the photomultiplier tube (PMT). A UV band pass filter (300 - 400 nm) located between the reaction cell and the PMT selectively transmits the light emitted by the SO/O₃ reaction. Efficient combustion in the ceramic tubes coupled with the UV band pass filter eliminates interference from non-sulfur containing analytes (e.g. nitric oxide, olefins, etc.) which also undergo chemiluminescent reactions with ozone. A background signal is typically present as a result of ozone-wall interactions and low level sulfur contamination of Detector gases. This background signal can be used as a troubleshooting aid (see Section 10).



- | | |
|--------------------------------|--------------------------|
| 1. Ozone Generator | 6. Vacuum Line |
| 2. High Voltage Transformer | 7. Particulate Filter |
| 3. Photomultiplier Tube Socket | 8. Pre-Ozone Restrictor |
| 4. Photomultiplier Housing | 9. Post-Ozone Restrictor |
| 5. Reaction Cell | 10. Pressure Transducer |

Figure 3 355 SCD Left Side



- | | |
|---------------------|-------------------------|
| 11. Amplifier Cable | 15. Fuses |
| 12. HV Cable | 16. Pressure Regulator |
| 13. PMT Amplifier | 17. Transfer line |
| 14. EMI Filter | 18. Front panel display |

Figure 4 355 SCD Right Side

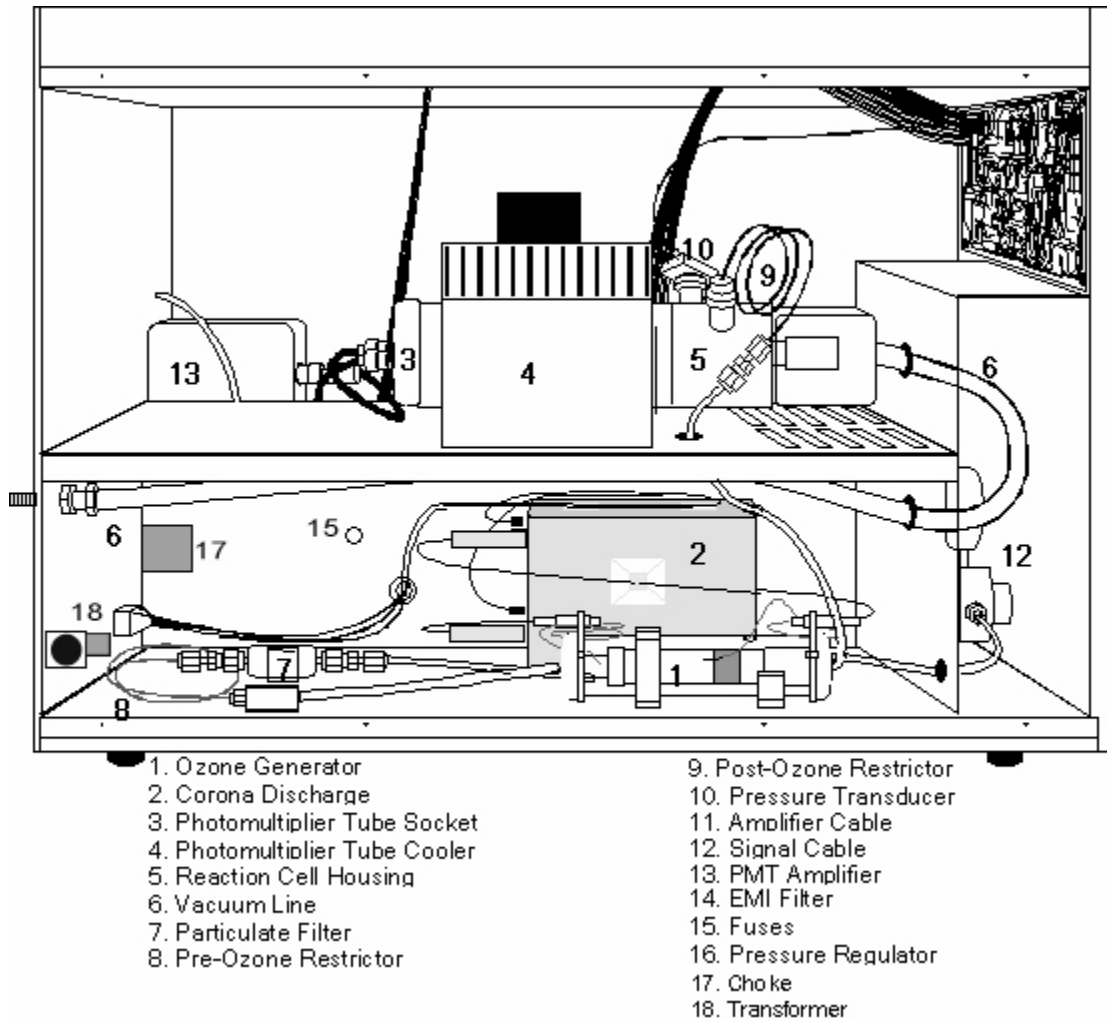
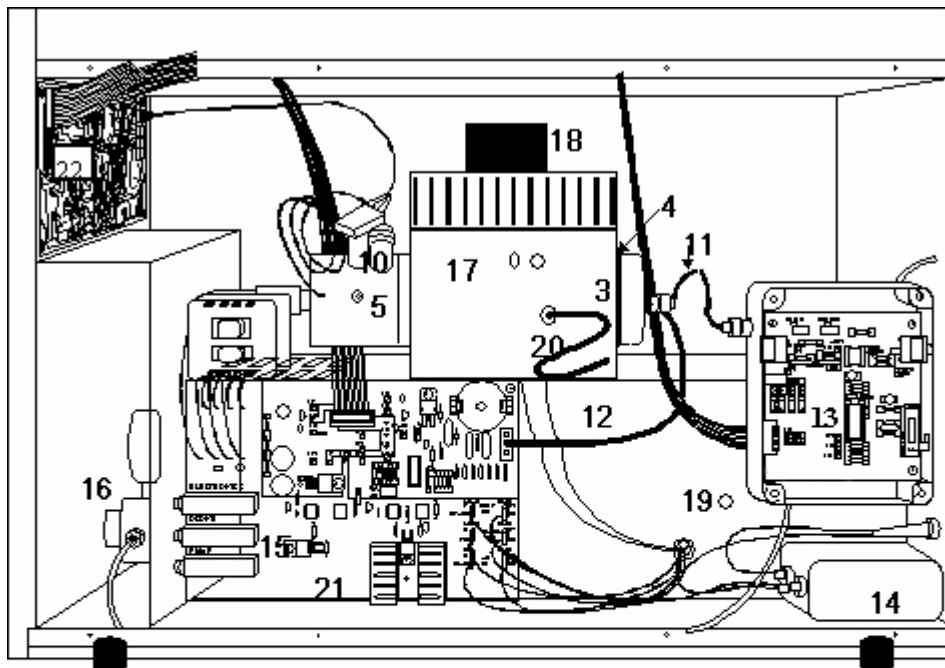


Figure 5 255 NCD Left Side View



- | | | |
|---------------------------------|---------------------------|-------------------------------|
| 1. Ozone Generator | 11. Signal Cable | 21. Power Supply Board |
| 2. Corona Discharge Transformer | 12. High Voltage Cable | 22. Front Panel Display Board |
| 3. Photomultiplier Tube Socket | 13. PMT Amplifier | |
| 4. Photomultiplier Tube Housing | 14. EMI Filter | |
| 5. Reaction Cell | 15. Fuses | |
| 6. Vacuum Line | 16. Pressure Regulator | |
| 7. Particulate Filter | 17. PMT Cooler | |
| 8. Pre-Ozone Restrictor | 18. Cooling Fan Regulator | |
| 9. Post-Ozone Restrictor | 19. Cooler Fuse | |
| 10. Pressure Transducer | 20. Thermocouple Wire | |

Figure 6 255 NCD Right Side View

Pressure Transducer

Vacuum in the reaction cell is measured using a pressure transducer. The pressure of the reaction cell can be monitored from the front panel and will typically range from 5 to 10 torr depending on the type of vacuum pump used, condition of the chemical (ozone) trap, ceramic tube position and the condition of the combustion chamber. The range of response is from 0 to 760 torr.

Vacuum Pump

There are two choices of vacuum pumps for the 355 SCD and 255 NCD. A two-stage, oil-sealed rotary vacuum pump is used to produce an operating pressure between 3 and 10 torr in the reaction cell. The oil-free Dry Piston pump produces a vacuum between 5 and 12 torr. All vacuum pumps serve the same purpose:

- Collection and transfer of the combustion gases from the Burner to the reaction cell.
- Transfer of ozone from the ozone generator to the reaction cell.
- Reduction of non-radiative collisional quenching of the emitting species in the chemiluminescent reaction cell.

The higher vacuum produced by the oil-sealed rotary pump facilitates a shorter residence time in the reaction chamber, and therefore reduces the incidence of collisional relaxation of the excited SO₂. This results in slightly lower detection limits using the oil-sealed pump as compared to the oil-free pump.

Chemical Trap

To protect the vacuum pump from exposure to ozone, a chemical trap is located at the inlet of the vacuum pump. This trap contains a catalyst that converts ozone to diatomic oxygen. This trap is a consumable part and should be replaced every 90 days of continuous Detector usage.

Oil Coalescing Filter

The oil-sealed rotary vacuum pump is operated with the gas ballast partially open to aid in the elimination of water produced in the Burner and transferred to the pump. As a result of the open gas ballast and the high flow rates of gases, oil vaporized in the pump can escape through the pump exhaust. To minimize oil loss, an oil coalescing filter is placed on the pump exhaust to trap vaporized oil and to return this oil to the vacuum pump oil reservoir. This is not necessary for use with the oil-free pump. A replaceable element in the filter is a consumable part and should be replaced every 90 days of continuous use.

FID Adapter (Optional)

The Agilent 355 Sulfur Chemiluminescence Detector is designed to mount into most GC detector ports and operate as a stand-alone sulfur detector. For some applications it is desirable to monitor both sulfur and hydrocarbon components using a single column without splitting. For this reason, Agilent offers a few adapter kits to mount the Dual Plasma Burner onto a Flame Ionization Detector for the simultaneous collection of FID and SCD chromatograms. During dedicated SCD operation, 100% of the column effluent passes through the Burner to the Detector. During simultaneous detection approximately 10-20% of the FID exhaust gases are drawn into the Burner through a restrictor, which reduces sulfur sensitivity to approximately 1/10 of the signal observed in a dedicated SCD Burner.

NCD Reaction Cell

The model 255 NCD reacts nitric oxide from the Burner and ozone from the Ozone Generator in the Chemiluminescence Reaction Cell. The reaction occurs

directly in front of the photomultiplier tube (PMT). A red cut-off filter between the reaction cell and the PMT selectively transmits the light from the nitric oxide and ozone reaction.

Efficient combustion in the ceramic tubes and the red cut-off filter eliminate interference from non-nitrogen containing analytes (sulfur dioxide, alkenes, olefins) that have chemiluminescence reactions with ozone.

A background signal is typically present as a result of ozone-wall interactions and low level nitrogen contamination from carrier and detector gases. The background signal is a useful troubleshooting aid.

NCD Photomultiplier Tube and Cooler

In the NCD, a red-sensitive PMT detects emissions from the nitric oxide and ozone reaction. A thermoelectric cooler cools the PMT 40 °C below ambient temperature to approximately -15 to -20 °C to reduce background noise from the PMT. The cooler operates whenever the Detector is connected to AC power.

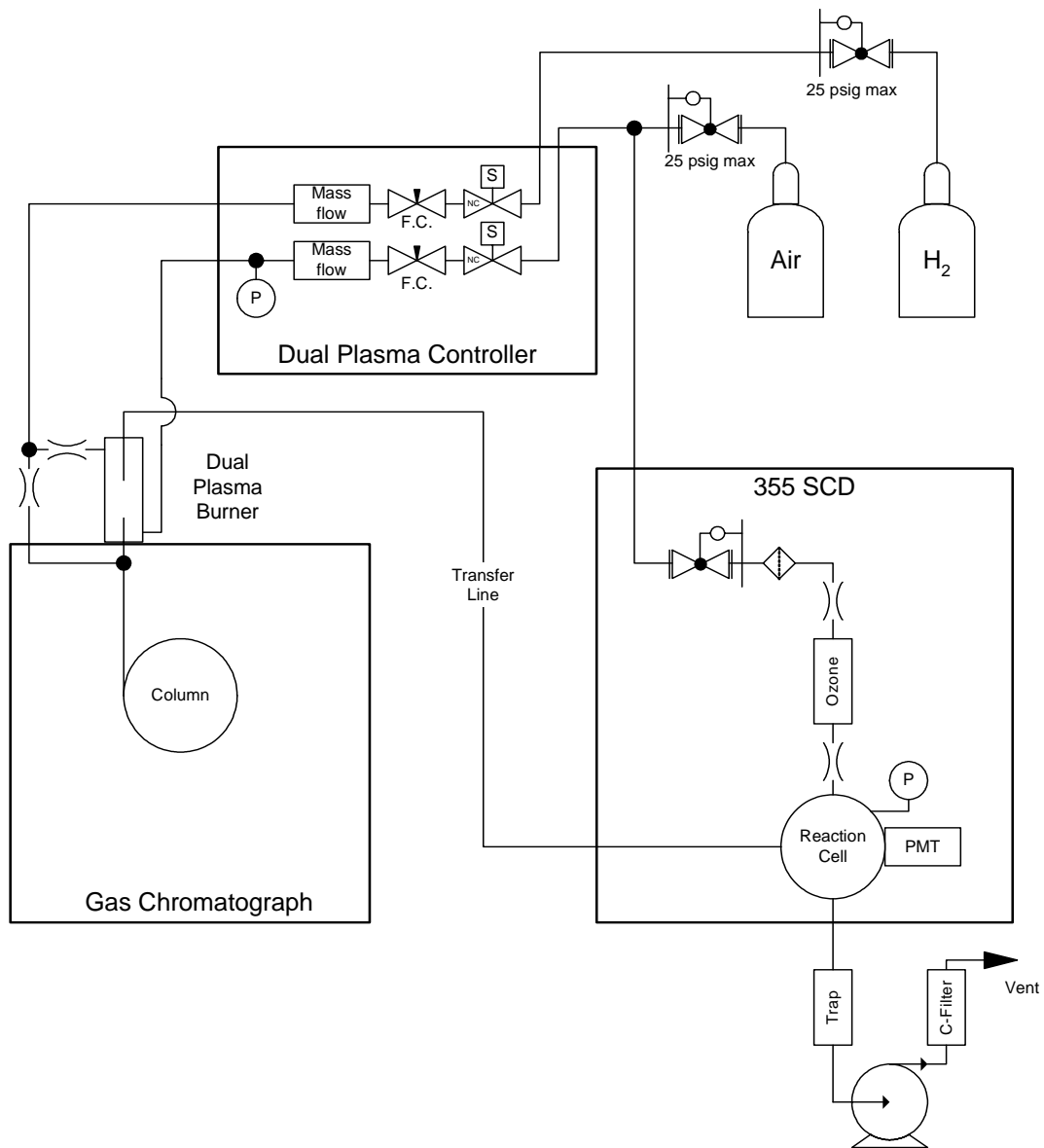


Figure 7 Schematic for 355 SCD

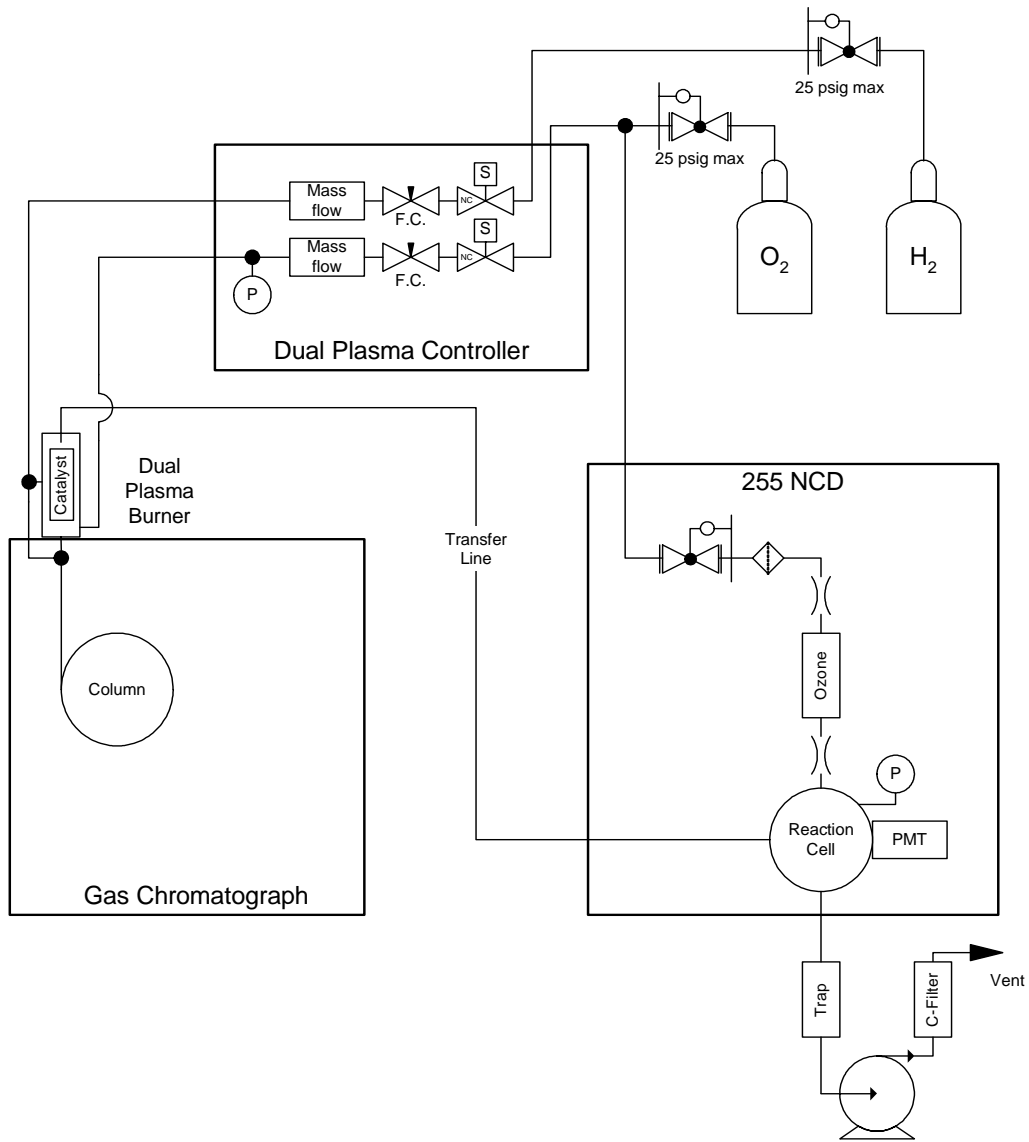


Figure 8 Schematic for 255 NCD, in Nitrogen Mode

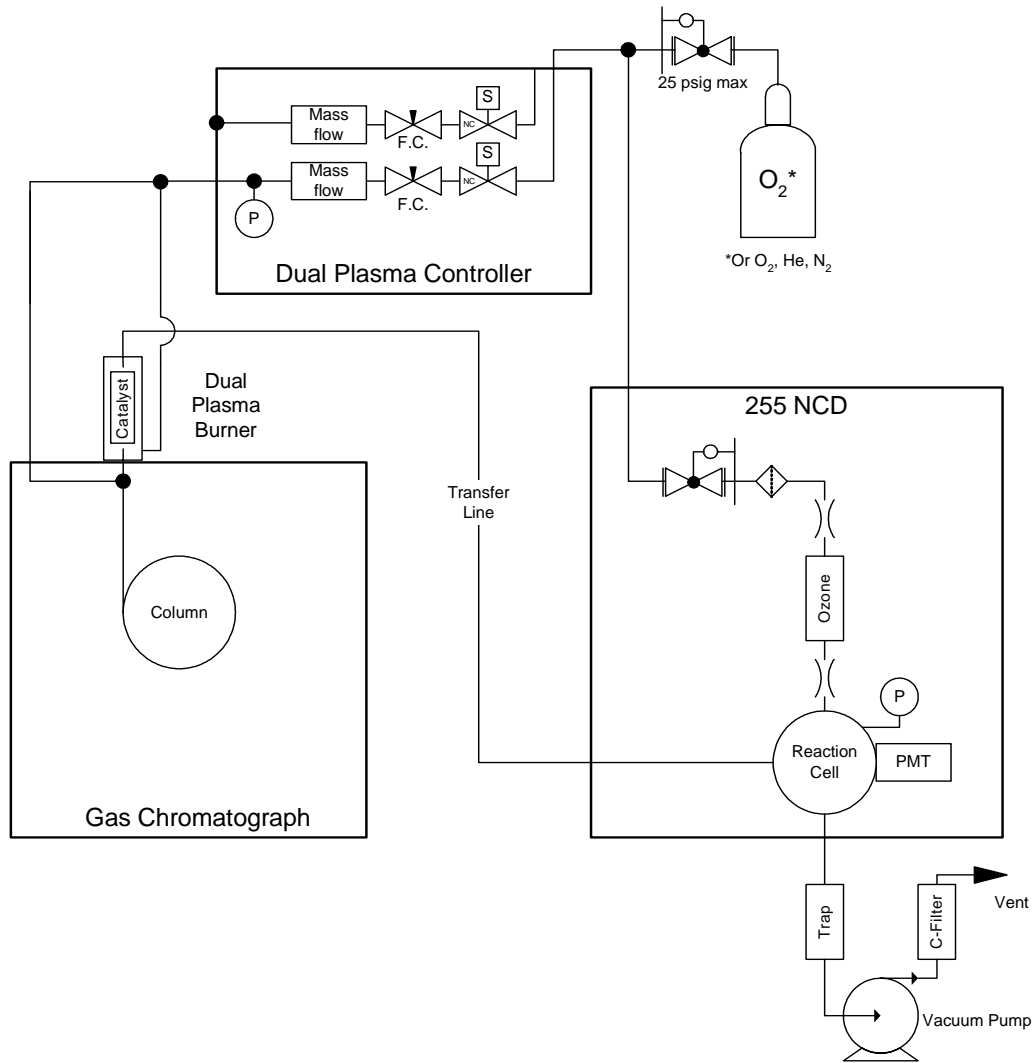
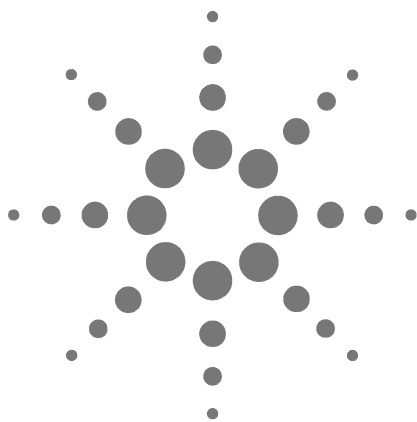


Figure 9 Schematic for 255 NCD, in Nitrosamine Mode



3 Installation

Overview	40
Step 1: Selecting a Location	41
Step 2: Unpack and Inspect the Instrument	45
Step 3: Set Up the Vacuum Pump	46
Step 4: Connect the Power Cord	56
Step 5: Install the Dry Compressed Air or O ₂ Supply	57
Step 6: Install the Signal Output Cables	58
Step 7: Install the Dual Plasma Burner	59
Step 8: Install the Dual Plasma Controller	60
Step 9: Install Column Connections	61
Step 10: Install the Transfer Line	62



Overview

Installation and start-up of the Agilent 355 SCD or 255 NCD by a qualified Agilent Service technician is recommended. If you choose to install the detector yourself, carefully read all of this chapter prior to installation of the instrument.

Although every reasonable safeguard against shipping damage has been taken, product damage may still occur due to excessive mishandling. If obvious damage has occurred during shipment, contact Agilent. Shipping materials for the 355 SCD or 255 NCD should be saved. If the instrument must be returned to the factory, it *must* be packed in the original carton to reduce the chance for damage during shipment. Replacement shipping containers can be purchased from Agilent.

WARNING

Substituting parts or performing unauthorized modification to the instrument may result in a safety hazard.

WARNING

Hydrogen is a flammable gas. Perform periodic leak tests to verify there are no leaks in the hydrogen lines and connections. Before making any connections, shut off the hydrogen supply. Connect or cap all fittings at all times when using hydrogen.

Step 1: Selecting a Location

The instrument should be placed on a clean, unobstructed surface approximately 22" (55 cm) deep by 10" (24 cm) wide that can support 44 pounds (19.9 kg) in addition to existing equipment. Figure 10 illustrates the relationship between the major system components. To facilitate proper heat dissipation, an additional 1-2" (2.5-5.0 cm) should be available at the rear and on both sides of the instrument. Distance between the SCD and the GC is limited by the length of gas and heater lines.

Position the Controller on top of the Detector box or in another convenient location near both the Detector and GC. The Controller requires supplies of hydrogen and oxidant (air for the SCD and oxygen for the NCD). The length of the gas inlet lines limit the distance between the Controller and the Burner to approximately 1 meter.

There are two pump options for the Detector, each with different size requirements. The Edwards RV5 oil-sealed vacuum pump requires a 6.3" (16cm) by 18.5" (47cm) area on a shelf or a nearby floor and a minimum height clearance of 22" (58 cm). The pump weight is 52 lbs (24 kg). The Dry Piston Pump (oil-free) requires a 9" (22 cm) by 14" (35 cm) area on a shelf or a nearby floor and a minimum height clearance of 12" (30 cm). The pump weight is 30 lbs (13.6 kg). The distance to the 355 Detector for both pump options is limited by the power cord connection—6 ft (2 m) for the oil-free piston pump or 8 ft (2.4 m) for the oil-sealed vacuum pump.

NOTE

Consider placing the oil-sealed pump over a plastic or metal container to capture any oil leaks or spills.

Power Requirements

See Chapter 2 for voltage and power requirements. Grounded outlets are required.

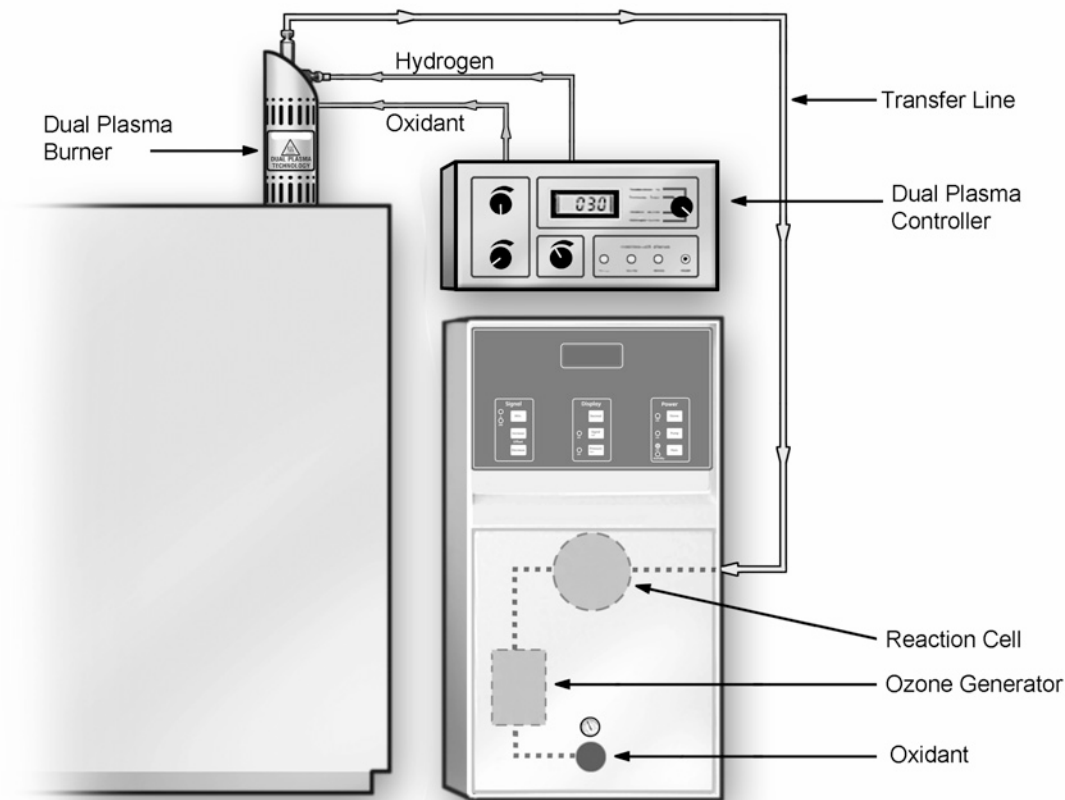


Figure 10 Drawing of the Detector with Dual Plasma Burner and Controller

Environmental Considerations

The instrument should be operated in an environment which is comfortable for human habitation with reasonably constant temperature and humidity. Operation of the instrument at elevated temperatures (>30 °C) may result in an increased background noise from the photomultiplier tube.

Combustion Gas Requirements

The Agilent 355 SCD and 255 NCD both require a hydrogen source; the 355 SCD requires an air source, and the 255 NCD requires an oxygen source. Oxygen may be used for the ozone generator in either Detector to obtain a modest increase in sensitivity; the Dual Plasma Burner was designed for use

with air as the oxidant in the SCD and oxygen as the oxidant in the NCD.

For the SCD, detector gases *must* be sulfur free (<1ppb) for proper Detector operation. In general, bottled air is preferred to “house” air from a compressor because compressors tend to generate lower quality air. In addition, pressure fluctuations induced by the air generator may be detrimental to Detector performance.

For the NCD and GC, use gases that are low in nitrogen and total hydrocarbons. Use gases with an "instrument" or "chromatographic" purity rating. Agilent recommends gases with a purity range of 99.995% to 99.9995%. Do not use oil-pumped supplied air since it contains large concentrations of hydrocarbons.

Use of appropriate traps on carrier, hydrogen and oxidant sources to improve support gas quality, such as a sulfur trap gas purifier, is recommended. The oxidant used to supply the ozone generator should be dry to prevent internal corrosion. Use of a moisture trap or dryer is recommended.

Two-stage pressure regulators

The combustion gases must be supplied to the Controller at a pressure of 25 psig or less. Use a two-stage pressure regulator rather than a single-stage regulator to eliminate pressure surges. High-quality, stainless-steel diaphragm-type regulators are recommended. On/Off valves at the regulators are useful but are not essential. Mount the valves at the outlet fitting of the pressure regulators.

If you use gases with a two-stage regulator up-stream, it will be sufficient to use single-stage regulators at the Detector.

Particle Filtration

Up-stream, inline particle filtration of 10 micron or better is required to prevent damage to Controller and Burner components.

WARNING

Ozone is a hazardous gas and a strong oxidant. Minimize exposure to ozone by using the instrument in a well-ventilated area and venting the exhaust of the vacuum pump to a fume hood. Turn off the ozone generator when the instrument is not in use.

Supply Tubing for Combustion Gases

Use preconditioned and cleaned copper or stainless steel tubing to supply gases to the 355 SCD and 255 NCD. Do not use ordinary copper tubing since it contains oils and other contaminants. Plastic tubing is not recommended since it is permeable to oxygen and other contaminants that can damage columns and detectors or cause elevated background. Teflon tubing may be acceptable in some clean environments.

WARNING

Secure all gas cylinders to an immovable structure or permanent wall. Store compressed gases in accordance with all safety codes.

WARNING

Wear eye protection when using compressed gas to avoid possible eye injury.

Venting Gases

During normal operation of the GC with the SCD, NCD, FID, other detectors and split/splitless inlet purge, some of the carrier gas and sample vents outside the GC. In addition, the vacuum pump will vent a small amount of ozone and other combustion products. If the components of the sample are toxic or noxious, vent the exhaust from the GC outlets to a fume hood. Also, vent the exhaust from the vacuum pump to a fume hood.

Step 2: Unpack and Inspect the Instrument

Before unpacking boxes, inspect them for signs of physical damage. If damage is observed, photographs should be taken in order to make a claim with the carrier should any equipment damage be found. Check contents of boxes against the shipping documents. Contact Agilent as soon as possible should a discrepancy be found.

Required Installation Tools

The following tools are recommended for installation of both the 355 SCD and 255 NCD:

- One adjustable wrench, with adjustment up to 1 inch (2.5 cm)
- One 1/4"-5/16" open-end wrench
- Two 3/8"-7/16" open-end wrenches
- Two 3/4"-9/16" open-end wrenches
- One 5/8" open-end wrench
- One 1/4" Nutdriver with hollow shaft (for FID restrictor)
- One Phillips head screwdriver
- One pair cotton gloves (for hand protection)
- One small flat-head screwdriver

Step 3: Set Up the Vacuum Pump

Initial connections require access to the rear of the Detector and the vacuum pump. Follow the installation instructions for either the RV5 Edwards pump or the oil-free dry piston pump, depending on your configuration.

WARNING

Exhaust gases from the pump should be vented to a fume hood to eliminate any potential hazard.

Installing the Edwards RV5 Pump Oil-Sealed Vacuum Pump

WARNING

The normal surface temperature of the pump body at ultimate vacuum (operation) at ambient temperature of 20 °C is 50 °C to 70 °C. If you use the pump at a high ambient temperature, the temperature of the pump body may exceed 70 °C, and you must fit suitable guards to prevent contact with hot surfaces. For more information refer to the pump operating manual.

WARNING

Do not operate the vacuum pump with the oil level below the minimum oil level mark or above the maximum oil level mark.

- 1 Install the chemical trap mounting bracket to the top of the pump with the screws and provided wrench. The chemical trap is a white plastic cylinder approximately 1 foot (30 cm) in length by 1.5 inches in (3.8 cm) diameter fitted with a barbed fitting on both ends (see Figure 11).
- 2 Remove the protective cap from the pump inlet port.
- 3 Install the metal conical screen and black centering o-ring into the pump inlet port (see Figure 13).
- 4 Attach the aluminum 1/2-inch barbed adapter to the pump inlet port with the metal clamping ring.
- 5 Slide the 2 inches Tygon vacuum hose over the pump inlet barbed adapter and secure with a hose clamp.
- 6 Slide another hose clamp over the 2 inches Tygon vacuum hose.
- 7 Remove the plastic caps from the barb fittings on the ends of the chemical trap.
- 8 Press the trap into the mounting bracket and the 2 inches of Tygon vacuum hose (see Figure 11 and Figure 12).
- 9 Tighten the hose clamp.
- 10 Locate the Oil Return Line Kit.

- 11 Remove the drain plug and the bonded seal from the oil mist filter. The bonded seal looks like a metal washer with a black inner o-ring.
- 12 Install the bonded seal to the oil mist filter drain adapter. The drain adapter looks like a drain plug with a small plastic nozzle.
- 13 Screw the black drain adapter and bonded seal into the oil mist filter.
- 14 Remove the plastic protective cover from the pump exhaust port. Place the centering o-ring on the pump exhaust port (see Figure 13).
- 15 Place the oil mist filter onto the o-ring on the pump exhaust port. Install the filter so that the gray half is above the white half of the filter (see Figure 11 and Figure 12).
- 16 Position the oil mist filter so the drain adapter points toward the gas ballast inlet (see Figure 11 and Figure 12).
- 17 Fit the clamping ring onto the adapter and oil mist filter and hand tighten.
- 18 Turn the gray plastic gas ballast knob counterclockwise to position II. Press the knob down against the spring and continue to turn counterclockwise until the knob is free. Remove knob from the pump. Confirm that the spring is still in place.
- 19 Locate the tall aluminum gas ballast control assembly, and install the small o-ring into the groove on the shaft.
- 20 Insert the gas ballast control assembly into the pump, pressing down against the spring, and turn it clockwise until the nozzle on the assembly is directly over the mark on the top of the pump.
- 21 Cut approximately 3/4 of the black silicone oil return line and insert the steel restrictor approximately half-way into the line. If needed, use a small screwdriver or other small tool to aid in positioning the restrictor.
- 22 Fit one end of the oil return line to the drain adapter on the oil mist filter. Fit the other end of the line to the nozzle on the gas ballast adapter. Ensure that the tubing is not tight and has no tight bends. Secure the line at each end, using the black hose clips provided.
- 23 Add oil to the pump via either of the two oil fill caps. The oil level should be between one-third and one-half when viewed in the oil sight glass. Replace the oil fill cap prior to operation of the pump.

WARNING

Do not operate the vacuum pump with the oil level below the minimum oil level mark or above the maximum oil level mark.

-
- 24 Place a hose clamp over the black heat shrink end of the 6' Tygon vacuum hose, and connect the hose to the barbed fitting labeled Exhaust on the back of the Detector. Tighten the hose clamp securely.
 - 25 The SCD should be placed near the GC and be accessible from the rear in order to connect the electrical power and the recorder signal cable.

- 26 The vacuum pump should be placed within approximately 3 feet of the Detector (elevation not important), in order to connect the vacuum hose from the back of the Detector to the chemical trap. A hose clamp should be placed over the hose, and the hose should be connected to the straight end of the chemical trap. Tighten the hose clamp securely.

WARNING

Vent the exhaust gas from the vacuum pump to fume hood to eliminate any potential hazard.

- 27 Place the vacuum pump in an adequately ventilated area or connect an exhaust line (not provided) to the outlet located at the top of the mist filter. Attach an aluminum 1/2-inch barbed adapter to the outlet with the metal clamping ring and another centering o-ring. Secure the exhaust line with a hose clamp (not provided) to the aluminum adapter. Route the exhaust line to a fume hood or other suitable discharge location.
- 28 Shape the exhaust line to capture excess water (see Figure 14). Do not allow water to condense in the exhaust line and drip back into the coalescing filter. Alternatively, install a water trap using a vacuum flask after the coalescing filter to capture excess water and prevent water from dripping back into the coalescing filter.

WARNING

It is imperative that water does not condense in the exhaust line and fall back into the mist filter. It is recommended that transparent 3/8 inch (0.95 cm) id tubing be used as an exhaust line.

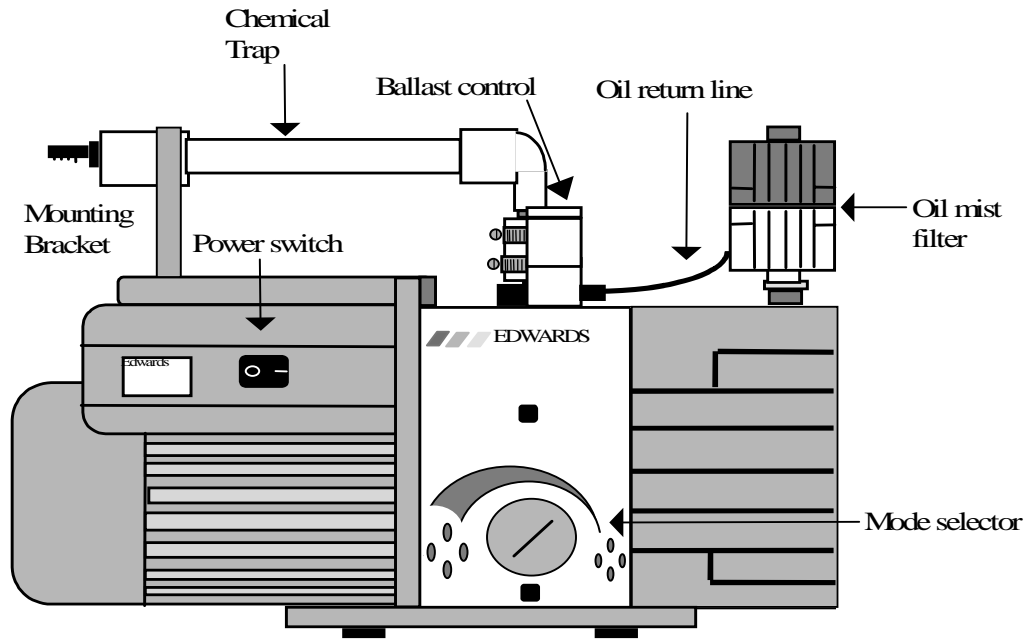


Figure 11 RV5 Oil-Sealed Vacuum Pump and Associated Traps (Front Side)

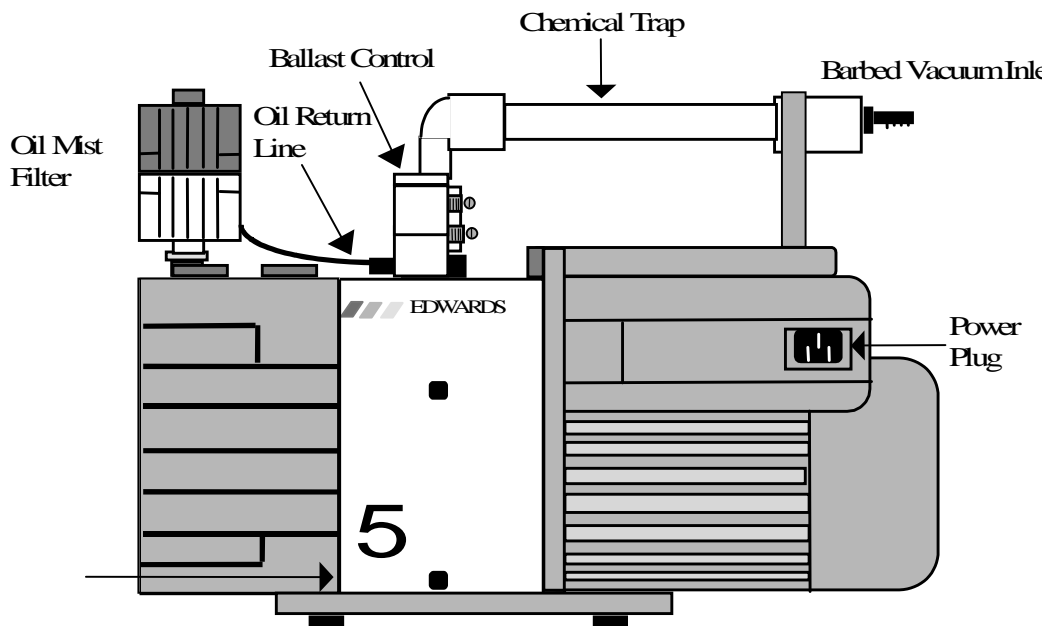


Figure 12 RV5 Oil-Sealed Vacuum Pump and Associated Traps (Back Side)

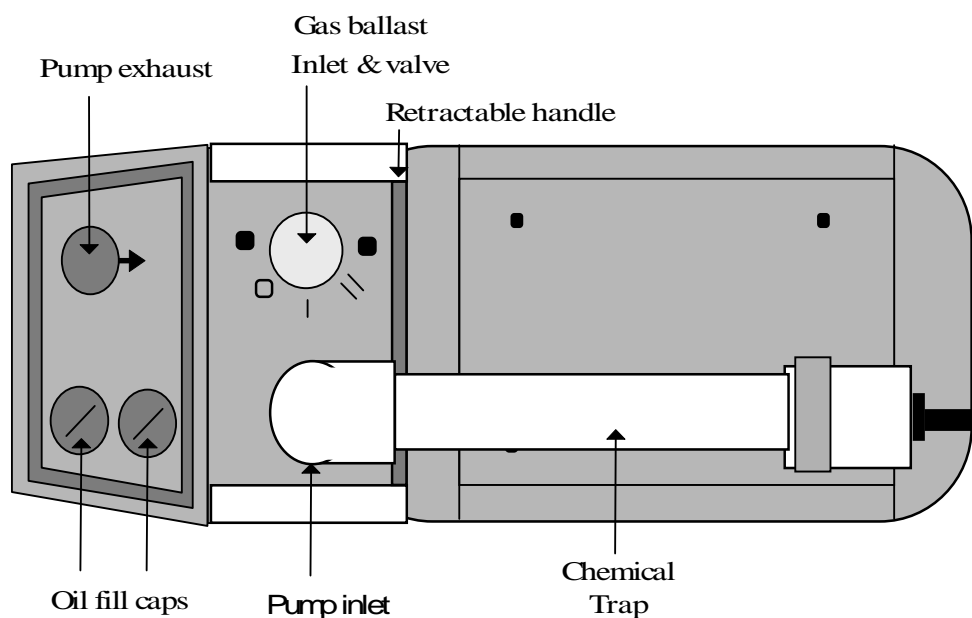


Figure 13 RV5 Oil- Sealed Vacuum Pump and Associated Traps (Top)

NOTE

Sometimes water condensation and accumulation are visible in the exhaust line. This is normal. However, do not allow water to continue to accumulate after approximately one week of operation. Significant water accumulation may indicate improper pump operation, an improperly vented exhaust line, or a Burner leak. Water accumulation in the exhaust line can cause damage to the pump, especially if allowed to fall back into the pump. Contact Agilent for advice if water accumulation continues to occur.

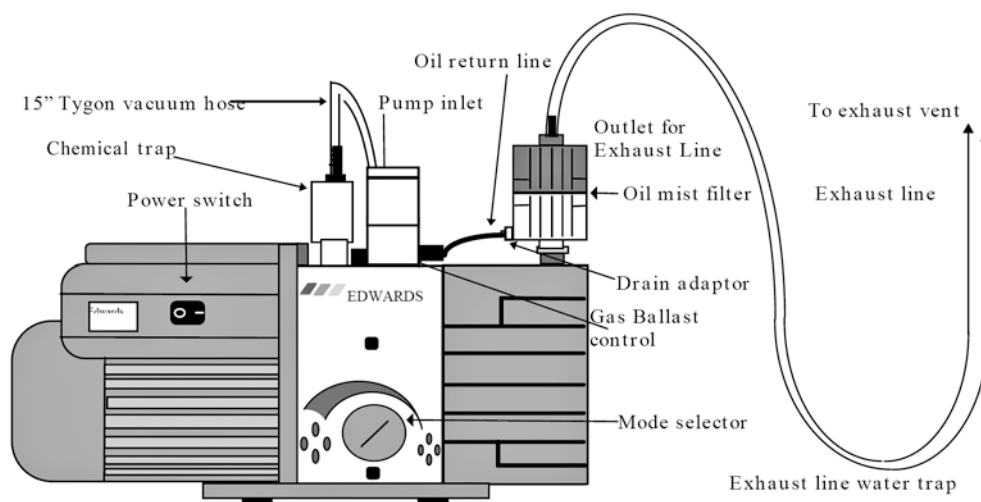


Figure 14 RV-5 Oil-Sealed Vacuum Pump Exhaust Line

Setting the Gas Ballast Position (RV5)

Set the mode selector halfway between the High Vacuum mode, the small **◆** symbol (see Figure 14) and the High Throughput mode. Do not set the mode selector to the High Throughput mode, the large **◆** symbol.

The RV5 vacuum pump and the Oil Drain Kit with ballast flow control ensure the vacuum pump operates continuously with a gas ballast flow. The purpose of the ballast control is to sweep ambient air into the pump oil. The air purges the water (created from the combustion of air and hydrogen in the Burner) and the oil (vaporized by the pump) into the oil coalescing filter. The filter separates the oil from the water, vents the air and water, and returns the oil to the vacuum pump.

The Oil Drain Kit with the ballast control continuously returns trapped oil in the oil mist filter to the vacuum pump. This feature reduces oil loss from the pump and minimizes the need to refill the pump with oil.

The Oil Drain Kit with the ballast control supplied is configured so that the gas ballast flow rate is equivalent to that with the gas ballast control on the pump in position II. For most applications, there is no need to change the gas ballast flow rate. If required, the gas ballast flow can be adjusted using the following procedure.

- 1 The restrictor plate on top of the aluminum ballast control has three screws. Remove the three screws that secure the restrictor plate. *Do not dismantle the assembly* (see Figure 16).
- 2 The restrictor plate has circular indentations. The position of the indentations with respect to the indentation on the side of the oil return assembly identifies the gas ballast flow setting. Turn the restrictor to the required position:

- To select no gas ballast flow (*not recommended*), turn the restrictor plate so that none of the indentations are aligned with the indentation on the side of the oil return assembly.
- To select low gas ballast flow, turn the restrictor plate so that the single indentation on the restrictor plate is aligned with the indentation on the side of the oil return assembly.
- To select high gas ballast flow (the setting the ballast control is shipped from the factory), turn the restrictor plate so that the two indentations are aligned with the indentation on the side of the oil return assembly.

Installing the Welch Dry Piston Vacuum Pump

The Welch Dry Piston Pump may be used as a direct replacement for an oil-sealed vacuum pump for the NCD or SCD. This pump produces all the advantages of an oil-free pump with little or no loss in instrument performance, however, operating Detector and Burner pressures will typically be a few Torr higher than those obtained with the oil-sealed pump.

To install the dry piston vacuum pump, follow these steps:

- 1 Remove the pump from its packaging and place on an accessible work surface with the power cord side nearest to you and the pump "INLET" port to your right. Refer to Figure 15 as a visual aid to these instructions.
- 2 Verify that the voltage of the pump matches that of the Detector.
- 3 Open the plastic bag containing the Dry Piston Pump Kit. Locate the brass elbow and brass barb (3/8" NPT). Remove their plastic protective caps and wrap three or four turns of Teflon tape (not supplied) onto the threaded connections. Screw the barb fitting into the elbow and tighten using wrenches. Similarly, remove the plastic cap from the pump's inlet fitting and screw elbow into it. Tighten the elbow so that the barb ends up pointed parallel to the floor and pointed toward the power cord side of the pump.
- 4 Locate the mounting brackets, 4 small screws and washers and spring clips. Use 2 screws and washers to attach the spring clips, one to each bracket.
- 5 Using the supplied Torx wrench, remove two Torx screws from the pump, the right corner screw located closest to you and the closest screw to you located immediately to the left of the pump's handle.
- 6 Attach the brackets to these locations using the Torx screws as showing in Figure 15.
- 7 Attach the short piece of 1/2" ID hose tubing supplied in the Dry Piston Pump Kit to the brass barb and use one of the supplied hose clamps to tighten it onto the barb.
- 8 Place a second hose clamp over the other end of the plastic tube. Insert the barb on the elbow end of a Chemical Trap into the open end of the short plastic hose that was just attached to the brass barb on the pump.
- 9 Rotate the Chemical Trap so that it is retained by the spring clips.

- 10 Tighten the hose clamp located on the plastic tube and the elbow barb end of the Chemical Trap.
- 11 Remove the plastic cap from the inlet end of the Chemical Trap. Taking the clear end of the vacuum hose and another hose clamp attach the vacuum hose to the inlet end of the Chemical Trap and tighten the hose clamp.
- 12 Attach the black end of the vacuum hose to the barb fitting located on the back of the Detector box and tighten the last hose clamp onto it to make this connection.
- 13 Using three or four wraps of Teflon® tape around NPT fitting of the supplied muffler or user supplied exhaust fitting to the pump's "EXHAUST" port. Any exhaust tubing attached to the pump exhaust should be positioned to avoid the accumulation of water from condensation of water vapor.

WARNING

Vent the exhaust from the vacuum pump to a fume hood or other exhaust system to eliminate any potential hazard.

- 14 Place the vacuum pump where it will be located when the pump is operated, within the length of the power cord to be attached to the female connector on the back of the Detector.
- 15 Make sure that the **On/Off** switch on the pump is in the on position.

Operation Notes (Welch Pump)

- 1 The pump is designed to start against atmospheric pressure. Therefore, if the pump is turned off inadvertently, it may be necessary to allow the Detector pressure to rise before restarting the pump.
- 2 The only user maintenance required is the periodic replacement of the piston sleeves and seals, e.g., after 8 months of continuous operation. Consult the pump's manual for further details.
- 3 Although, the pump is not damaged by exposure to ozone, use of the Chemical Trap is highly recommended. Refer to the pump manual and seal replacement kit instructions for more detailed information.
- 4 High line voltage will cause the pump to overheat and trip its thermal fuse.
- 5 When new, or after changing seals, the pump may require a short break-in period (2-4 hours).

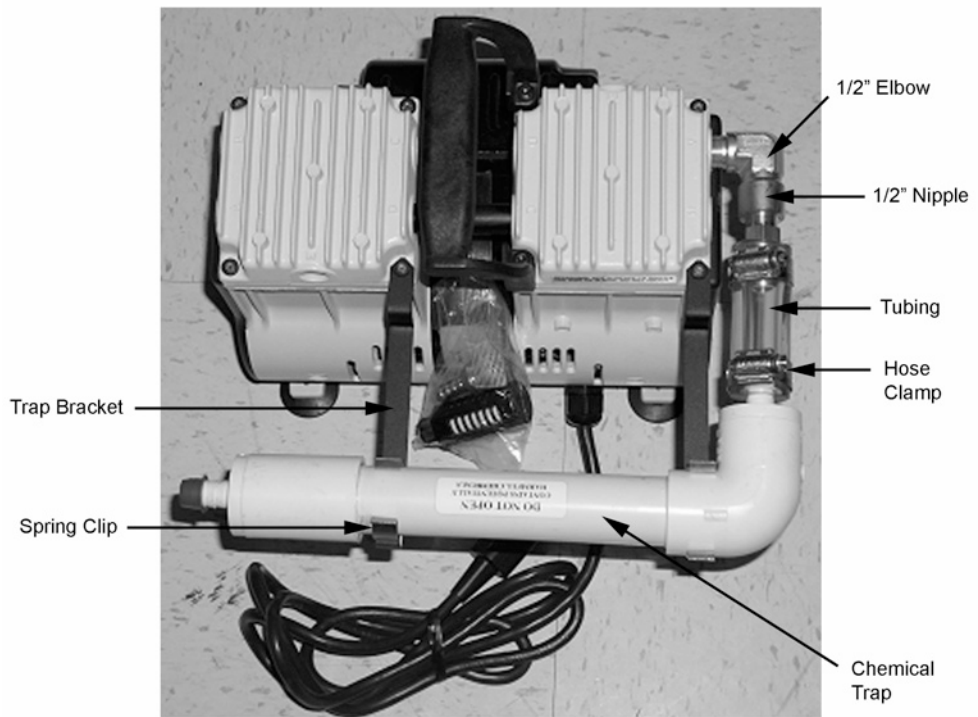


Figure 15 The Welch Dry Piston Pump

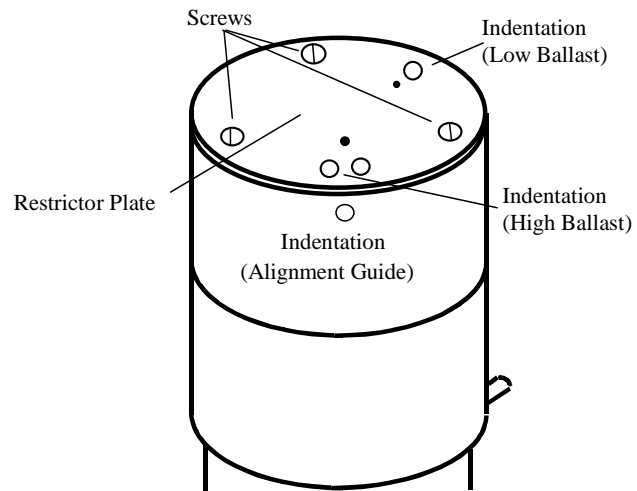


Figure 16 Oil Drain Kit with Ballast Control

6 Turn the switch on the vacuum pump to the On position.

Note the position of the oil level in the window after operating the pump for several hours. For the next several days of operation, recheck the oil level daily. If the oil level is increasing, water is accumulating in the oil reservoir. Confirm that the water vapor is properly being expelled from the oil mist filter. If the oil level is dropping, excess air flow through the pump is forcing oil out of the vacuum pump. Turn the restrictor plate so that the single indentation on the restrictor plate is aligned with the indentation on the side of the oil return assembly. After adjustment of the gas ballast, allow the system to operate for an additional day and check the oil level again.

It is normal for the oil to appear foamy from air in the oil when viewing the oil in the oil level window. The purpose of the ballast control is to sweep ambient air into the pump oil. Ensure the oil level when the pump is operating is not above the “Full” mark on the pump.

Step 4: Connect the Power Cord

Connect the pump power cord to the female socket on the back of the Detector (see Figure 17). The pump has an **On/Off** switch located on the electrical motor and this switch should be turned On. *Do not* connect to the AC power supply at this point in the installation procedure.

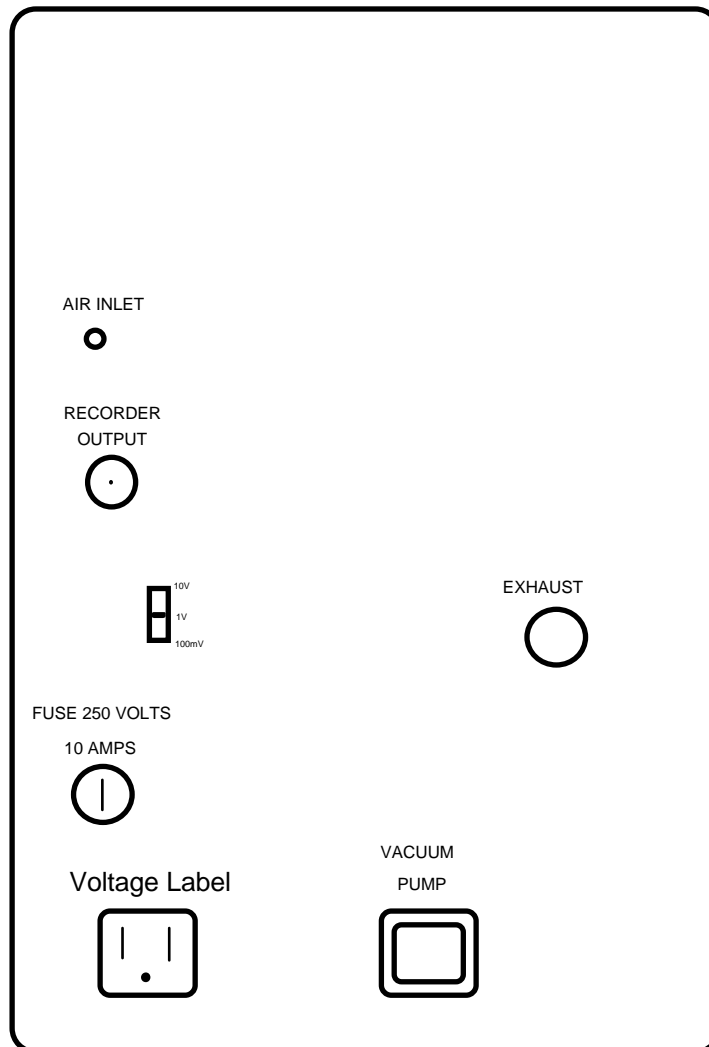


Figure 17 SCD (230 V Unit) Rear Panel Diagram

Step 5: Install the Dry Compressed Air or O₂ Supply

Connect a 1/8" OD Teflon (PFA) line fitted with a 1/8" brass Swagelok nut from the AIR INLET at the rear of the Detector (see Figure 17) to a supply of dry compressed air or oxygen. The air regulator located inside the front door of the Detector should be set to approximately 3-6 psi.

Step 6: Install the Signal Output Cables

Signal output cables are available from Agilent as standard equipment and can be used with most data systems. Confirm that the output cable supplied is correct for your system. A standard cable fitted with two crimp lug connectors is supplied for use with most integrators, recorders or data systems. Attach the BNC connector end of the recorder cable to the matching output connector, labeled RECORDER OUTPUT, on the back of the SCD (see Figure 17).

Standard Cable Connection

The standard recorder cable is connected to the integrator by installing the red crimp-lug connector to the signal terminal (+) and the black crimp-lug connector to the negative terminal (-). No additional ground connection is required.

HP 3390 Series Integrator Cable Connection

The keyed-edge connector on the HP 3390 series cable is attached to the signal input of HP 3390 series integrators. Note that the connector can only be installed one way.

HP 3396 Integrator Cable Connection

The jack plug on the HP 3396 cable is attached to the analog signal input connector at the rear of the integrator. This cable also works with the Agilent 35900 controller.

HP 5890 GC Analog Input Board

A cable made specifically for this input board is available. This board is used when the SCD signal is input to ChemStation.

Agilent 6890 GC Analog Input Board

A cable made specifically for this input board is available. This board is used when the SCD signal is input to ChemStation.

Step 7: Install the Dual Plasma Burner

Remove the cover plates from the Detector area of the GC to expose the hole into the oven through which a Detector is normally mounted. If the GC has more than one available Detector position, pick the most convenient one.

Prepare the GC by cutting the inside liner and top liner per Figure 17. Note that mounting fastener patterns will vary by GC manufacturer. Make sure the notch in the inside liner is on the right, in order to accommodate the geometry of the Burner as it sits in the shroud and mounting plate.

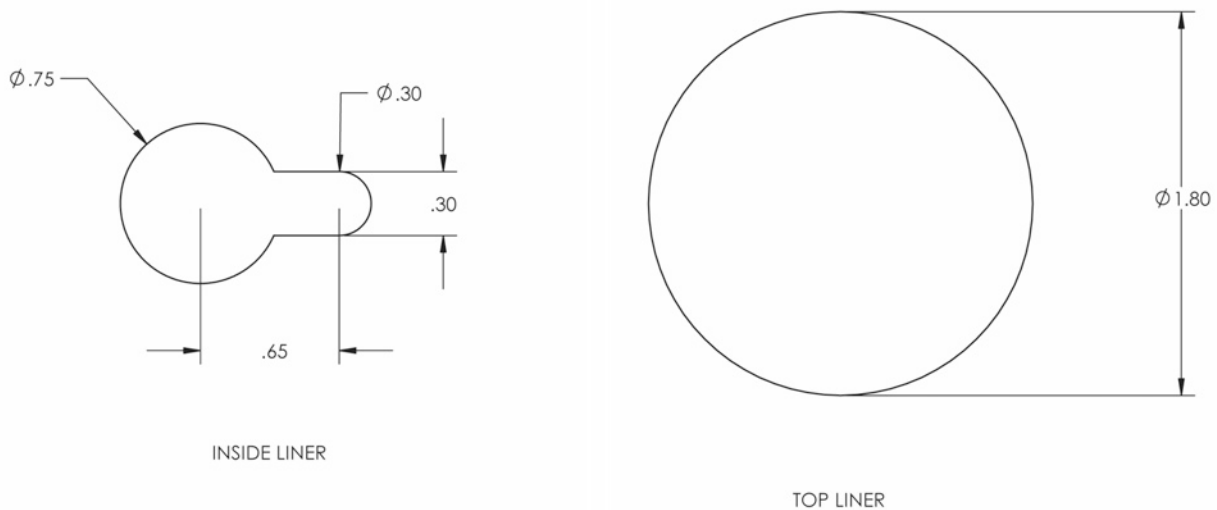


Figure 18 Dimensions of GC Liner Cut-Outs

Align the Burner mounting plate with the mounting screw holes on the GC. Clear the hole into the GC oven of interfering insulation, and then secure the mounting plate onto the oven with the screws provided. Attach the Burner's heated base connector to the GC's temperature control circuit. Consult your GC's operation or service manual to confirm proper connection of the 100 ohm RTD sensor and the cartridge heaters.

NOTE

Occasionally geometric design changes will occur within or between GC models. If the mounting plate provided does not match up with the top of the GC, contact Agilent for additional information.

Position the Dual Plasma Burner (column end down) into the tapered fitting of the heated base, with the lower hydrogen line and pin aligned with the slot in the heated base. The Burner should be secure when properly positioned.

Step 8: Install the Dual Plasma Controller

Position the Controller such that the gas lines from the Burner can be easily attached to the back of the Controller. Connect the Controller to both a hydrogen source and an oxidant source, per Step 1. Connect the gas supplies to the 1/8" bulkhead unions marked "Oxidizer Inlet" and "Hydrogen Inlet." Clean copper tubing (1/8" OD) is recommended.

Connect the two 1/16" gas delivery lines (provided) from the Burner to the appropriate fitting on the rear of the Controller marked Oxidizer or Hydrogen in the Outlets (to Burner) area. Use a 1/4" open-end wrench to tighten the nut onto the bulkhead unions, using a 9/16" wrench to back-up the union.

Connect the yellow thermocouple plug from the Burner to the thermocouple jack at the rear of the Controller. The thermocouple connector will only fit one way, with the iron terminal (the one with the two grooves) down. Connect the Burner heater line to the two pin locking connector on the back of the Controller.

The Controller can be used for either 100/115 or 230 VAC. The correct voltage for your Controller is set at the factory; however, this should be verified upon installation. The voltage selection is shown through a small window on the power entry module (see Figure 19). If the voltage setting is incorrect, check to make sure that the proper fuses are installed. Contact Agilent for a replacement fuse set.

WARNING

Do not change the voltage selector without changing the main power fuse. Operating the Controller box without the proper fuse may damage the electronics.

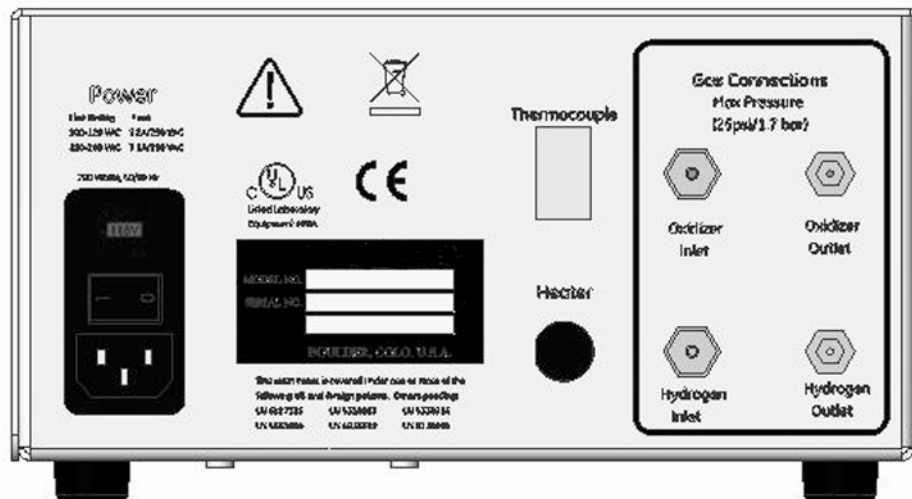


Figure 19 Dual Plasma Controller Rear Panel

Step 9: Install Column Connections

The Burner operates under reduced pressure and there will be a slight vacuum on the end of the column. If a higher outlet pressure for the column outlet is desired, fused silica capillary restrictors may be attached to the end of the analytical column (both capillary and packed) prior to making the Detector connection.

Capillary Columns

Place the column nut over the end of the capillary column. Place the appropriate fused silica adapter ferrule onto column. Remove a few centimeters from the end to remove any particles that may have entered the column. Insert the GC column into the Burner by 108-109 mm from the upper end of the nut (114-115 mm if measured from the flat bottom of the nut). Do not force the column. The pathway is narrow and may take several tries to seat it correctly. Tighten the column nut finger tight, or until sealed using a 7/16" open-end wrench to back-up the hydrogen inlet fitting to prevent it from slipping.

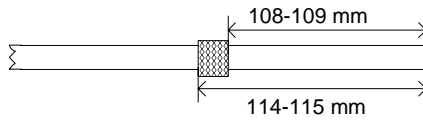


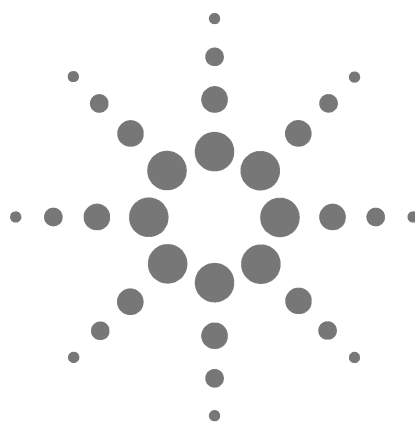
Figure 20 Measuring Column Insertion

Packed Columns and Columns with an Outside Diameter > 0.8 mm

Connect a short piece (0.5 m or less) of deactivated fused silica tube, for example 0.32 mm internal diameter, to the Detector end of the column. Follow the procedure for capillary column connection as above.

Step 10: Install the Transfer Line

Connect the black transfer line (extending from the side of the Detector) to the top connector on the Burner and tighten with a 3/8" open-end and 7/16" open-end wrench (backing up the union on top of the Burner to prevent its position from slipping).



4 Front Panel Controls and Initial Startup

Detector Front Panel Controls 64

Detector Interface Setup 69



Detector Front Panel Controls

As illustrated in Figure 21, the front panel is divided into three sections: signal control, display output control, and power control. Each section is described below. All the front panel LEDs are red in the ON mode and darkened in the OFF mode except for POWER. This LED toggles green for ON and red for STANDBY. In STANDBY mode, the front panel display and high voltage to the photomultiplier tube (PMT) are turned off. The red LED in the STANDBY mode serves as a reminder that power is still supplied to the instrument.

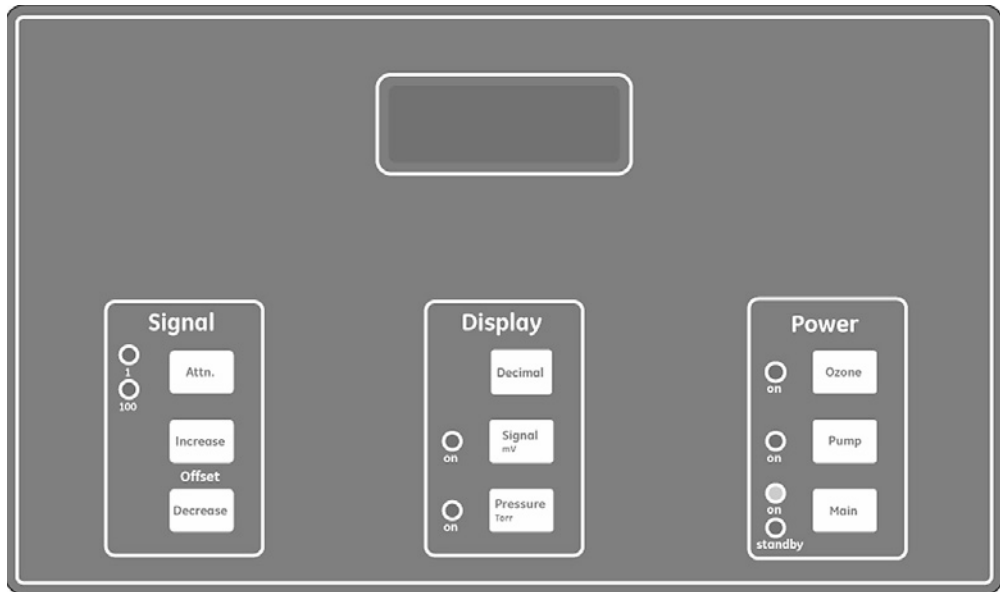


Figure 21 Front Panel Controls

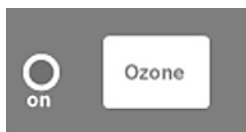
Power Controls



ON: Turns on front panel displays and high voltage to the PMT.
STANDBY: Darkens panel and turns off high voltage to the PMT. **Does not shut off main power.**



Turns pump ON (red) and OFF (dark).
Functions in both ON and STANDBY modes.



Turns ozone generator ON or OFF.
Functions in both ON and STANDBY modes if the pump is producing sufficient vacuum.

Display Output Controls



Controls the range of the front panel display between high sensitivity (0.1-200mV) and low sensitivity (0-2V). **Does not** affect recorder output signal.



Displays the output signal of the SCD in millivolts. (Note: Changing the output range of the SCD by adjusting the recorder switch on the back panel **does not** change the range of the display.)



Displays the pressure of the chemiluminescence reaction chamber in Torr (mm Hg)

Signal Controls

The SCD and NCD use analog amplifier circuitry for the measurement of the current produced by the photomultiplier tube. Two Front Panel Controls are used to adjust the output signal of the Detector.



Changes the gain of the amplifier by a factor of 100. For high sensitivity measurement of sulfur or nitrogen compounds (measurement of low levels of sulfur or nitrogen compounds), the attenuation should be operated in the 1 position. For samples containing high levels (ppm to percent) of sulfur or nitrogen compounds, use the 100 position.



The baseline signal from the SCD and NCD can be adjusted from 0 to $\pm 1\%$ of the full scale recorder output using the offset control. (e.g., +10 mV to -10 mV for a 1 V full scale setting). This control can be used to offset the background signal.

Dual Plasma Controller Controls

The Agilent Dual Plasma Controller provides easy access to basic settings.

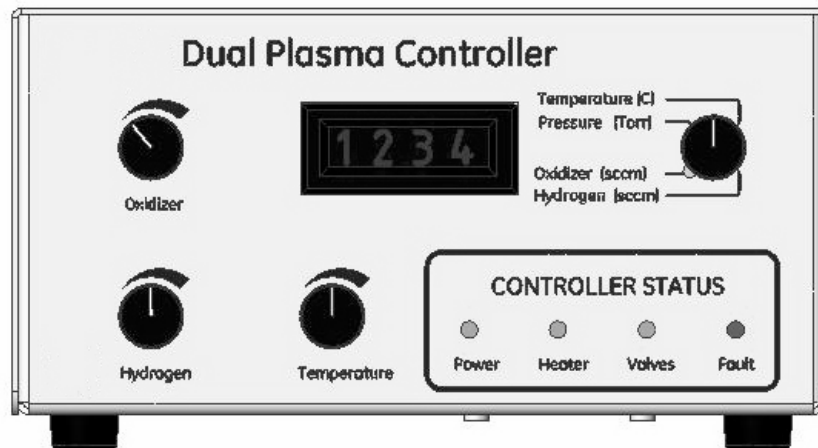


Figure 22 Dual Plasma Controller Front Panel

Oxidizer and Hydrogen Control Knobs	Allow you to adjust the oxidizer and hydrogen flow rate.
Temperature Control Knob	Allows you to adjust the Burner temperature.
Selector Control Knob	Changes the display to show the current value or set point for each setting (temperature in $^{\circ}\text{C}$, pressure in Torr, oxidizer in sccm, or hydrogen in sccm).

Controller Status
Lights

Illuminate to reflect current status. The **Power** LED indicates power is on; the **Heater** LED indicates temperature set point has been obtained; the **Valves** LED indicates the solenoid valves open; and the **Fault** LED indicates a drop in pressure or excessive temperature (see Chapter 7, “Troubleshooting” for details).

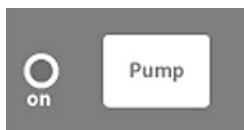
Initial Startup

Vacuum Test

With the 1/8" Valco cap on the black PFA transfer line still in place and the air supply to the ozone generator off, set the internal air regulator to 0 psi (fully counterclockwise), and power on the SCD or NCD by plugging the power cord into the house power supply. The SCD and NCD will power up in STANDBY mode, with the pump and the ozone generator OFF. For this test, the desired mode is power ON, vacuum pump ON, ozone generator OFF, display range in the low sensitivity mode and PRESSURE signal displayed. Press the following controls in the order shown in order to start the vacuum pump and to view the reaction cell pressure.



Press until green ON LED is lit



Press until red LED is lit



Press to display reaction cell pressure in torr

After the pressure has stabilized (~5 minutes), record the pressure below. Typical pressure in the reaction cell should be 1-2 torr with the oil-sealed pump, or 5-8 torr for the oil-free pump.

Reaction cell pressure, with air to ozone generator OFF (0 psi setting on internal regulator) and transfer line capped: _____ torr

Tighten connections if necessary and check to make sure pressure stabilizes in the expected region. If proper pressure is not obtained contact Agilent for assistance. If the reaction cell pressure is within the expected range, record the value, reset the internal air regulator to 3-6 psi, turn the pump OFF and proceed with the recorder test.

Recorder Test

The standard 355 SCD and 255 NCD recorder output configuration is 1 volt full scale. In addition, an output range can be set with the recorder output selection switch located on the back panel (Figure 11). Use a small screwdriver to adjust the switch to the proper position for your integrator or data system.

To check that the recorder cable has been properly connected to the integrator or data system, set the integrator to a high sensitivity setting (e.g. attenuation of x 1) and plot the background signal. Use the Output Offset to decrease the Detector baseline. If the recorder cable is connected correctly, the baseline will shift in response to changes in the Output Offset. If the polarity of the connection is incorrect, a negative response will be observed when the baseline is increased, and vice versa. Switch the polarity of the wires to correct this problem. If no response is observed, re-check the signal connections and repeat the test. Contact Agilent if there is no data system response after completion of this test.

Detector Interface Setup

Initial Checkout

Careful attention to eliminating leaks in the Detector interface will lead to better Detector sensitivity and easier troubleshooting if problems develop.

- 1 Check that the gas connections have been made correctly and that they are tight.
- 2 Plug the 3-prong connector for power on the Controller into a 100-volt, 50/60 Hz, 115-volt, 50/60-Hz or 230-volt, 50/60 Hz AC outlet.
- 3 Connect the oxidant delivery line to the oxidant inlet nut on the Burner. Leave the heater cable disconnected and the thermocouple plugged in.
- 4 Connect the black PFA transfer line from the Detector to the top of the Burner (see Figure 1 and/or Figure 2).
- 5 Turn on the Controller, the Detector power, and the vacuum pump.
- 6 Let the system pump down to a stable pressure (~5 minutes).
- 7 Read the pressure from the LCD display on the Controller. It should be <30 torr, with higher pressures observed for packed or megabore columns due to the higher column flow. Record this pressure for reference.

Pressure of Burner with column flow but without hydrogen and oxidant flow to Burner:_____ torr

The reaction cell pressure on the Detector should be less than 5 torr (10 with oil-free pump).

- 8 If pressure readings are not within the above specifications, then there are leaks in the system. Leak check all positive pressure points (swage nuts, tees, crosses) with SNOOP or a leak Detector. Tighten fittings if necessary and recheck the pressure. Confirm that the Burner fittings (oxidant inlet nut, tee, and hydrogen nut) are strongly hand-tightened. If necessary, tighten the gas delivery lines and transfer line. If pressure readings are still high, contact Agilent for service.

WARNING

Do not overtighten! Overtightening the fittings on the ceramic tubes can cause the tubes to break. Vespel ferrules that are greatly distorted by overtightening cannot be used again.

- 9 Turn off the Controller and the vacuum pump.
- 10 Reconnect and/or turn on the hydrogen and oxidant supplies to the Controller.

Adjust the hydrogen and oxidant inlet pressures to 25 psig (1.7 bar).

Refer to Table 1 on page 75 for typical operating conditions.

Monitoring Oxidizer and Hydrogen Flow with the Dual Plasma Controller

Hydrogen will flow to the Burner only if the temperature is above 325 °C and the pressure of the Burner is <575 torr.

Turn ON the vacuum pump at the Detector and the power to the Dual Plasma Controller. The power LED should illuminate. When the temperature reaches approximately 325 °C, the valve's LED should illuminate, indicating hydrogen and oxidant flow.

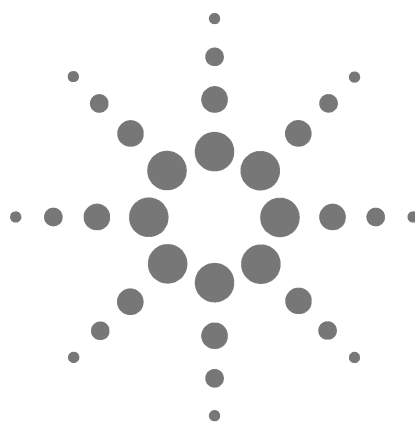
Use the Controller display knobs to select display of oxidizer or hydrogen flow.

WARNING

Hydrogen gas is explosive and must be handled carefully. Keep away from sources of ignition.

WARNING

The Burner is hot. Do not touch the Burner. Let the Burner cool before performing any operations involving the Burner.



5 Operation

Start-Up Procedure	72
Detector Operation	73
Typical Operating Conditions	75
Detection Limits	76
Instrument Shut-Down	77
Special Operating Modes	78

Start-Up Procedure

- 1 Turn on the GC and set the carrier flow rate.
- 2 Turn on the hydrogen and oxygen to the Dual Plasma Controller.
- 3 Turn the Detector from STANDBY to ON.
- 4 Press the PUMP button until the red LED illuminates. Make sure the vacuum pump is running.
- 5 Turn on the oxygen to the ozone generator and set the regulator inside the front door of the Detector to between 3 and 6 psig.
- 6 Allow the system to evacuate for approximately 1 minute before turning on the ozone at the front panel. A properly functioning system will show a background signal deflection between the ozone on and ozone off.
- 7 Turn ON the Dual Plasma Controller using the ON/OFF switch on the rear panel of the Controller. As the Burner heats, the SCD or NCD background will increase and then slowly decrease. The hydrogen and oxidant flow will automatically start when the temperature of the Burner rises above approximately 325 °C.
- 8 Check the LCD display of Dual Plasma Controller to confirm that all pressure, temperature, and signal responses are within the desired specifications.

WARNING

The Burner is hot. Do not touch the Burner. Let the Burner cool before performing any operations involving the Burner.

Detector Operation

Detector Stability and Response

The time required for system stabilization varies depending on the application, system cleanliness, presence of active sites and other factors. Useful results could be generated within 30 minutes of start-up, especially with a previously operated system. A longer stabilization time is likely to be required upon changing critical system components, such as the combustion tubes or the GC column. In addition, gas flow rates may drift initially as thermal equilibrium is reached due to changes in gas viscosity with temperature. Therefore, it is good practice to monitor gas flow rates and adjust them accordingly.

Even though a system may not be fully stabilized, sample injections can be made within minutes of instrument start-up. Whether the results are useful largely depends on application. Typically, an elevated baseline will initially be observed, which will diminish upon successive programmed runs.

After stabilization has been reached, the system should exhibit good short-term and long-term precision. Of course this also depends on the application and concentration of components being measured. As an example, analysis of thiophene in benzene at the 1 ppm sulfur level yielded 1.4% RSD (n=10) over about 2 hours and 3.6% RSD (n=42) over about 96 hours. As expected, carbon disulfide at a lower concentration of 90 ppb sulfur yielded 2.6% and 10.4% RSD, respectively.

Column Bleed

Accumulation of column bleed causes silicon dioxide to build up in the Burner. This silicon dioxide creates active sites that are detrimental to performance. In many cases, the choice of column can be optimized for a particular application. Column bleed can be minimized by the use of oxygen traps on the carrier gas, low-bleed columns, and lowest possible maximum oven temperature.

Coking

Contamination from some sample matrices can reduce sensitivity. Crude oils containing volatile metal complexes may contaminate ceramic tubes. The incomplete combustion of certain hydrocarbon-containing compounds leaves behind coke deposits on the tubes. Coke deposits may be removed from the Burner by reducing the hydrogen flow rate. The Dual Plasma Burner is much less susceptible to coke formation than other designs.

Hydrogen Poisoning

Hydrogen poisoning of the ceramic tubes occurs when there is no oxidizer flow through the ceramic tubes. The result is extremely reduced, or no response. Hydrogen poisoned tubes can not be reconditioned and should be discarded.

Contaminated Gases

The use of clean gases for the 355 SCD is essential for optimal performance. High purity gases (99.999% pure or better) are advised. Sulfur and other contaminants from gases may accumulate in the column and bleed out over time desensitizing the tubes and causing elevated baselines. The use of sulfur traps is highly recommended for all gases.

Fluctuating Pressures

Fluctuations in pressure, especially from gas generators, will affect Detector response. It is therefore recommended that only bottled gases equipped with a dual stage regulator or appropriate steps to ensure stable pressure supplies are used.

Typical Operating Conditions

The Controller is calibrated at the factory for flow rates to deliver gas in sccm units. The following table summarizes the typical operating conditions:

Table 1 Typical Operating Conditions

Condition	SCD	NCD
Detector Pressure (Torr)	4-8 (6-12 [*])	4-10
Dual Plasma Controller Pressure (Torr)	300-400	100-250
Burner Temperature (°C)	800	900-950
Hydrogen Flow Rate (sccm)	40-50	4-6
Oxidant Flow Rate (sccm)	60-65 (air)	8-12 (oxygen)
Background Noise (mV)	0.3-2.0	0.3-1.0

* Oil-free pump
Thermocouple lifetime at 950 °C is diminished.

The recommended conditions should yield satisfactory results for most applications and should be used as a typical starting point for any method development. Like any detector, however, there are optimum conditions which may vary somewhat from the recommended conditions. In optimization of conditions for the Dual Plasma Burner and Controller the following guidelines should be considered:

High flow rates of hydrogen and oxidant can release enough heat at high temperature to vaporize combustion tubes and cause blockages downstream where the materials condense. High flow rates will eventually cause the pressure in the Burner to exceed its fault cut-off limit of about 600 Torr. For this reason, do not exceed the recommended flow rate by more than about 25%.

A higher hydrogen to oxidant ratio may initially show higher response but later yield a reduced response because of the accumulation of contaminants, such as soot or other active species, that reduce the Detector response.

Operating the Burner at higher temperatures will place more demand on the heater, thermocouple and seal materials, effectively shortening their lifetime.

In general, when making any parameter change, keep in mind that the system may require time to reach equilibrium.

Detection Limits

The following table lists the detection limits which can be expected for typical chromatographic conditions, assuming proper operation of the Detector and chromatographic systems.

Table 2 Expected Detection Limits for Chromatographic Conditions

Type of Injection	Volume	Column	Detection Limit Per Compound as Sulfur
Liquid, Split 1:10	1 μL	Capillary ≤ 0.32 mm ID	50 parts per billion
Liquid, Split 1:100	1 μL	Capillary ≤ 0.32 mm ID	0.5 parts per million
Liquid, On-column or splitless	1 μL	Capillary ≤ 0.32 mm ID	5 parts per billion
Liquid, Splitless	10 μL	Capillary ≤ 0.32 mm ID	0.5 parts per billion
Gas, Split 1:10	1 cm^3	Capillary ≤ 0.32 mm ID	50 parts per billion
Gas, Direct on-column	1 cm^3	0.53 mm ID	5 parts per billion
Gas, Direct on-column	1 cm^3	Packed	5 parts per billion
Gas, Direct on-column	10 cm^3	Packed	0.5 parts per billion

Instrument Shut-Down

Daily Shutdown

- 1 Toggle off the ozone generator.
- 2 Turn off the air regulator (counter-clockwise), located inside Detector door.
- 3 Toggle power to "stand-by."
- 4 Leave the vacuum pump and Dual Plasma Controller operating at all times.

Complete Shutdown

- 1 Toggle off the ozone generator.
- 2 Turn off the air regulator (counter-clockwise), located inside Detector door.
- 3 Toggle power to STANDBY.
- 4 Turn off power to the Dual Plasma Controller.
- 5 After 15 minutes, toggle off the vacuum pump, so the Burner cools and moisture is removed from the system.
- 6 Turn off gases to unit.

Special Operating Modes

Using the 255 NCD in Nitrosamine Mode

By default, the 255 NCD is configured to detect nitrogen. To change from nitrogen to nitrosamine mode, first turn off and unplug the Controller. Remove the cover, find jumper P6 and the positions labelled High Setpoint and Low Setpoint located on the printed circuit board near the left front of the Controller. Move the jumper position from the High Setpoint to the Low Setpoint position. This changes the temperature control range to 350-500 °C to be used for nitrosamine analysis. Refer to see Figure 9 on page 37 for a schematic drawing of the 255 NCD in nitrosamine mode.

Hydrogen is not used in the nitrosamine mode. Turn off and disconnect the hydrogen inlet gas. Using the supplied tee, couple the oxidant outlet with the two Burner inlets. Use of oxygen is recommended at 5-10 mL/min because the Burner can easily be cleaned by raising the Burner temperature to about 900-1000 °C in flowing oxygen. Alternatively, helium or argon can be used, however, these gases will not permit in situ cleaning at elevated temperature.

The presence of catalyst in the quartz combustion tube will generally yield the highest sensitivity for nitrosamines. However, for some sample types that contain potentially interfering nitrogen compounds, such as nicotine, it may be desirable to remove the catalyst to obtain better selectivity. This is achieved by removing the quartz combustion tube and using a straight 1/16" rod or tube to push the catalyst out of the tube (save the catalyst for reuse or recovery as it contains 90+% platinum). Replace the combustion tube and optimize the burner temperature as desired (in general it is desirable to increase the pyrolysis temperature by 50-100 °C when the catalyst is not used). Refer to the "Tube Replacement for the NCD" on page 102 for additional instruments with regard to tube removal and replacement.

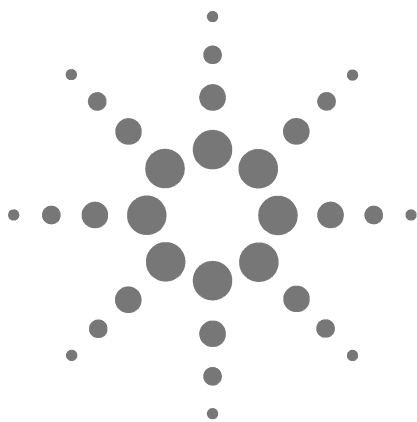
Using the SCD in High Sensitivity Mode for Nonhydrocarbon Gaseous Samples

There are circumstances in which it may be desirable to operate the SCD in a non-typical manner. These could include the analysis of very low levels of sulfur species in a nonhydrocarbon gas matrix, such as helium, carbon dioxide, or even hydrogen. It should be possible to measure low ppb or high ppt levels of sulfur species.

In the case of a hydrogen matrix, the sample matrix itself can suffice as the lower source of hydrogen for Dual Plasma operation in a non-chromatographic mode. For instance, a flow rate of nominally 20 SCCM of the sample, a hydrogen calibration gas and a "clean" hydrogen source can be alternately introduced to the Burner. The sample and calibration gas would be introduced into the normal column connection. The side port of the splitter fitting would be plugged and the clean hydrogen source would be plumbed to the lower hydrogen inlet port. The upper hydrogen and air flow rates would be adjusted to nominally 30 and 65 SCCM, respectively.

In the case of nonhydrocarbon gaseous samples, the potential for coking is not a concern, so a Dual Plasma is not necessary. Some improvement in sensitivity

can be achieved by eliminating the lower plasma. This is accomplished by plugging the side port of the splitter fitting and teeing the air into the lower hydrogen line, much like the configuration used for Nitrosamine analysis as shown in Figure 9. A slightly lower air flow rate, such as 40 SCCM and a hydrogen flow rate of around 60 SCCM is used. Sensitivity of around 0.1 pg S/sec or less should be readily achievable. It is not possible to cover all potential applications, so some optimization may be desirable.



6 Maintenance

Pump Maintenance	82
Cleaning the Detector	83
Changing the Oil Mist Filter (RV5)	84
Reaction Cell Cleaning	85
Flow Sensor Calibration	88
Detector Sensitivity	89
Assembling the Dual Plasma Burner for Component Replacement with the SCD	90
Assembling the Dual Plasma Burner for Component Replacement with the NCD	95
Tube Replacement for the SCD	98
Tube Replacement for the NCD	102



Pump Maintenance

To maintain optimum performance of the Agilent 355 SCD and 255 NCD, routine replacement of the chemical trap (for ozone destruction), oil coalescing filter and oil (Edwards oil-sealed pump only) is necessary. Refer to Table 3 for the expected life span of each replacement part or material.

It is beneficial to keep a maintenance log that tracks when maintenance is performed and any instrument or operational changes that might impact performance. Also keep track of detector flow rates (oxidizer and hydrogen), pressures (burner controller and reaction cell in detector), and background signal (the difference between ozone “on” and ozone “off”).

Table 3 Operating Life of Components for Edwards RV5 Vacuum Pump

Component/Material	Operating life*
Chemical Trap (RV5)	~ 3 month
Oil Coalescing Filter (RV5)	~ 3 months
Pump oil†	~ 3 months

* The operating life is based on the total time logged during operation of the Detector with the Burner and the ozone generator ON.

† Pump oil can be purchased from a supplier or directly from Agilent: SAE 10W-30, Multiviscosity Synthetic Motor Oil such as, MOBIL 1 or AMSOIL.

Cleaning the Detector

You can clean the external housing of the Detector with a damp cloth using water or non-abrasive cleaners. Turn off power to the Detector and disconnect it from main power prior to cleaning. Do not spray liquids directly on the Detector. Wipe dry with a clean, soft cloth.

No cleaning agents which could cause a hazard as a result of reaction with the Burner unit are to be used in cleaning the instrument.

Contact Agilent to address concerns about the compatibility of specific cleaning agents with the Burner unit.

Changing the Oil Mist Filter (RV5)

The oil mist filter on the RV5 pump has two components: the charcoal odor filter and the oil coalescing filter element. To replace the filters, disassemble the oil mist filter assembly with the 4 mm long-handled allen wrench (provided). The smaller charcoal odor filter sits on top of the larger oil coalescing filter element. It is recommended to replace the oil coalescing filter element after 90 days of continuous use, however replacement of the charcoal odor filter is optional. After replacing the filter, re-assemble the filter assembly and attach it to the pump flange.

Reaction Cell Cleaning

Over time, the reaction cell and UV pass filter (SCD) or IR pass filter (NCD) will develop a build-up of material which should be removed for optimum sensitivity. The cleaning schedule depends upon Detector use and the nature of the analyses; however, it is recommended the cell should be cleaned annually. The cleaning procedure requires removal of the photomultiplier tube (PMT) from the Detector, and special precautions are required to prevent damage to the PMT.

Exposure of a photomultiplier tube to bright light can result in damage to the PMT, even when the high voltage is off. To avoid potential damage, minimize light exposure. The black PMT cover included in the Detector accessories package is recommended for this procedure. Carefully read all the instructions below before attempting to remove the PMT. If you have any questions regarding this procedure please contact Agilent.

To clean the cell and pass filter:

- 1 Disconnect the main power cord (if the vacuum pump is operated from an independent power outlet, unplug the power to the pump as well).

WARNING

Any operation requiring access to the inside of the equipment, could result in injury. To avoid potentially dangerous shock, disconnect from power supply before opening the equipment.

- 2 Remove the right side panel from the Detector and disconnect the high voltage and coaxial signal cables from the rear of the PMT housing (See Figure 3 and Figure 15).
- 3 Turn off all room lights and minimize outside light sources.
- 4 Unscrew the PMT socket connector assembly. Carefully pull the socket assembly and the PMT out toward the rear of the instrument at a slight angle to remove the PMT/socket assembly from the housing.
- 5 Immediately place the black PMT cap over the PMT, being careful to avoid placing fingerprints on the PMT window. Do not remove the PMT from the socket assembly. After the PMT cap is in place, carefully place the PMT and socket assembly on a soft surface inside a drawer or other dark location to minimize exposure of the PMT to light. The room lights may be turned on at this point.

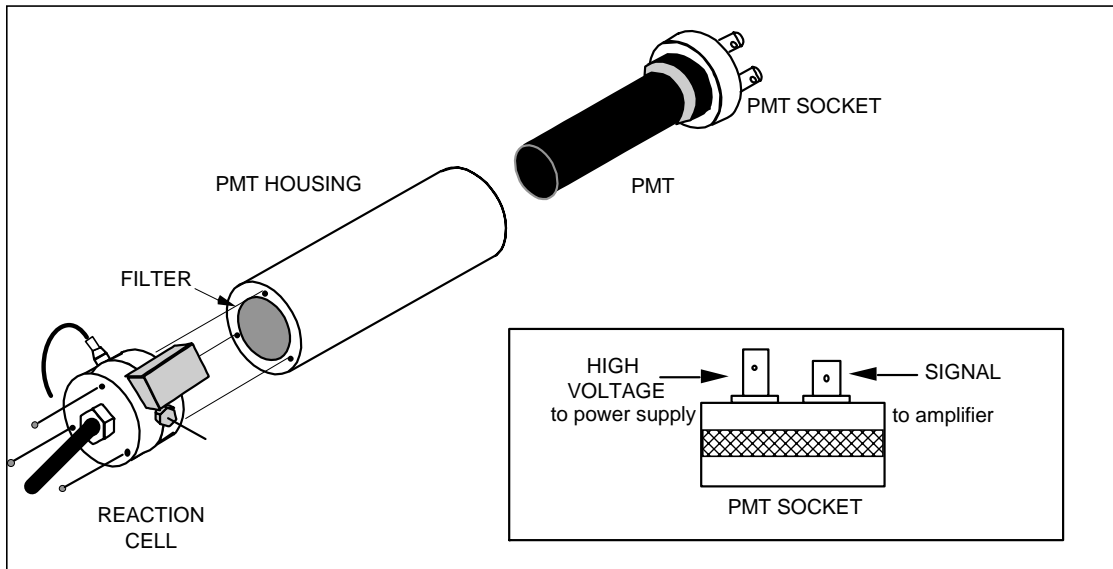


Figure 23 Reaction Cell, PMT Housing and PMT Socket

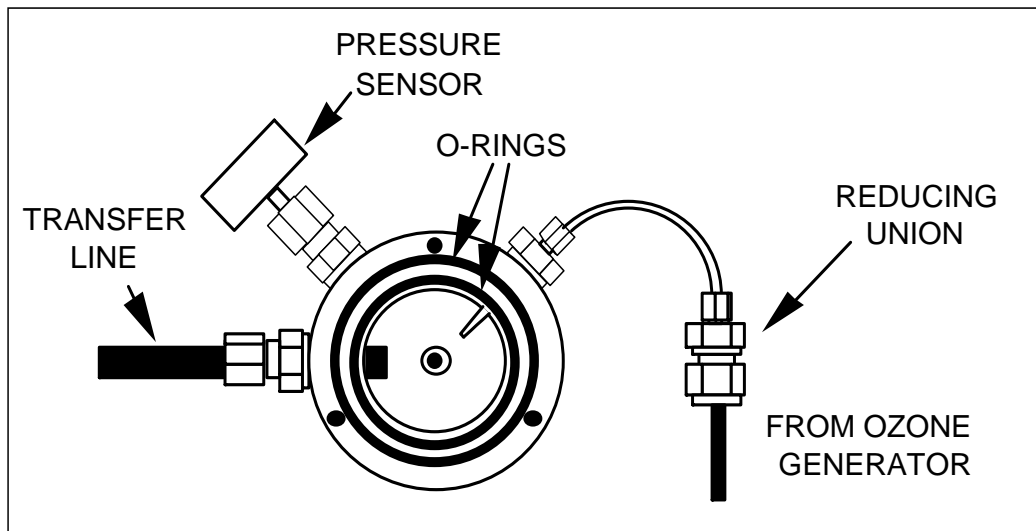


Figure 24 Reaction Cell

- 6 Use a 7/64" Allen wrench to remove the three mounting screws from the reaction cell. Slowly pull the reaction cell back from the PMT housing. The optical filter is located between the reaction cell and the PMT housing. Remove the optical filter from the housing by tipping up the back of the Detector, if necessary, and allow the filter to fall onto a soft cloth.
- 7 Inspect the o-rings and replace them if they show any wear.

- 8 Clean any deposits on the optical filter using a soft cloth or Kimwipe dampened with methanol or deionized (DI) water. Do not leave fingerprints or fibers on the cleaned filter. Deposits inside the reaction cell can be cleaned in the same manner, however, care must be taken to avoid bending the ozone inlet tube that extends into the cell.
- 9 Reseat the pass filter into the PMT housing. Confirm that the two o-rings around the reaction cell are properly seated in the O-ring grooves (see Figure 24). Carefully re-align the reaction cell to the PMT housing, making sure the o-rings remain properly seated, and secure the cell to the housing with the three screws. (The two o-rings must be correctly seated in order to obtain a vacuum-tight and light-tight seal.)
- 10 To re-install the PMT, minimize all light sources. Remove the PMT cap, being careful not to touch the PMT window. Insert the PMT and the socket assembly into the PMT housing and screw in the socket assembly until it is seated tightly against the housing. The room lights may now be turned on.
- 11 Reconnect the PMT high voltage cable to the MHV connector (longer connector) and the PMT signal cable to the BNC connector (shorter connector) at the back of the socket assembly (see Figure 23).

WARNING

The high voltage and signal cables must be attached to the proper connectors on the pmt socket; damage to the pmt will occur if the cables are not properly connected.

- 12 While the side panel is still removed, reconnect the AC power cord to the Detector and switch the front panel power to ON. To ensure that the o-rings have been properly sealed, monitor the Detector baseline at "attenuation 1" for several minutes (with the room light ON), and after noting the baseline signal, turn the room lights OFF. If the baseline signal significantly decreases, the o-rings are incorrectly positioned and the PMT must be removed and the cell re-installed.
- 13 If no change in the baseline is observed, check the system for vacuum leaks by turning on the vacuum pump and monitoring the pressure as described in Section 7. If the pressure readings are comparable to start-up values, the o-rings are properly positioned. If a significantly higher pressure is observed, the PMT must be removed and the cell re-installed.

Flow Sensor Calibration

The hydrogen and oxidant flow sensors installed in the Dual Plasma Controller have very good repeatability, but significant non-linearity. They have each been factory calibrated at the midrange flow rate (50 SCCM) using an NIST traceable flow meter. Over the typical operating range for the SCD, the flow sensors should produce accuracy of better than $\pm 10\%$ of reading. However, due to sensor non-linearity at low or high flow extremes, error greater than this could be observed.

If greater accuracy is desired at a particular flow rate range, it is possible to re-calibrate the flow sensors. A reference flow meter, appropriate gases, and a trim-pot adjustment tool are required for this procedure. With the controller on and no gases flowing, the zero calibration points are set with RP6 and RP4 for the hydrogen and oxidant flow streams, respectively. Connect a reference flow meter to the gas outlet(s) on the back of the controller. Connect pressurized gas line(s) to the inlets on the back of the controller. Connect jumper (JP1) to bypass the pressure fault circuitry. Allow the burner temperature to exceed $325\text{ }^{\circ}\text{C}$ in order for the hydrogen and oxidant valves to be open. The spans of the sensors are then adjusted for the particular flow stream to the desired flow rate as measured with the reference flow meter. The span adjustments are set with RP3 and RP2, for hydrogen and oxidant respectively, so that the display on the controller matches the value obtained with the reference flow meter. Upon completion, remove the reference flow meter and return the bypass jumper (JP1) to its original position.

Detector Sensitivity

Detector sensitivity is an indicator reflecting the performance characteristics of a given system, and is a useful tool to determine when Detector maintenance is warranted. Sensitivity is typically reported as a minimum detection limit (MDL) as calculated from the following formula:

$$MDL = \frac{0.66 \times (Am'tN / S) \times (PktoPkNoise)}{(Wd_{1/2ht}) \times (Signal)}$$

Where Am't N/S (amount of nitrogen or sulfur) is the mass of nitrogen or sulfur in picograms that reaches the Detector, PktoPkNoise (peak-to-peak noise) is the measure of the noise (e.g. in mV), Signal is the height of the peak in the same units, and Wd_{1/2ht} is the width of the peak at half height in seconds. The constant 0.66 is used in the calculation assuming the MDL S/N = 3.29.

Before it is released from the factory, each 355 SCD must pass an MDL level of <0.5 pg Sulfur/second and each 255 NCD must pass an MDL level of <3.0 pg Nitrogen/second. The response from individual detection systems may vary by a factor of 2 or 3; however, it is typical for Detectors to perform in the 0.1-0.3 pg S/second range for the 355 SCD and in the 1-2 pg N/second for the 255 NCD when tested at Agilent.

Assembling the Dual Plasma Burner for Component Replacement with the SCD

The following procedure can be used to assemble the Dual Plasma Burner for use with the SCD or for replacement of Burner components, such as ceramic tubes. Refer to Figure 1 on page 26 for proper part nomenclature.

- 1 Slide the 0.066" internal diameter (I.D.) double taper ferrule onto the lower Burner tube. The tube should extend approximately 2 mm past the end of the ferrule.
- 2 Insert the lower Burner tube and double taper ferrule into the Burner inlet fitting.
- 3 Slide the 1/4" Burner adapter over the top of the lower Burner tube all the way down to the Burner inlet fitting and screw it onto the fitting finger-tight.

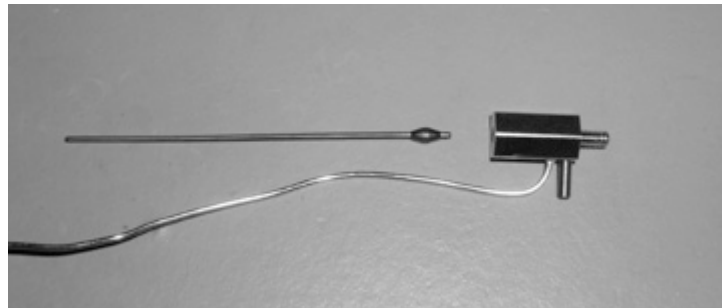


Figure 25 Ferrule Placement on Lower Burner Tube

- 4 Slide a 1/4" Swagelok nut over the Burner adapter and then slide a 1/4" ferrule over the Burner adapter and position it into the 1/4" nut. Note: If a graphite ferrule is used, a small amount of shavings may be created and some graphite will be left on the tube surface; this is normal. Avoid allowing any shavings to fall inside a tube.
- 5 Center the lower Burner tube so that it will slide into the tapered union. Insert the lower end of the tapered union fitting into the 1/4" Swagelok nut and screw it on finger-tight. If necessary, the brazed H₂ line can be gently bent out of the way, however, be careful not to stress the brazed (welded) connection.
- 6 Insert the large ceramic tube into the quartz heater assembly. Position a 1/4" ferrule (flat end butted up against the top of the swivel nut) onto the large ceramic tube. With the ferrule positioned against the swivel nut, approximately 0.5 cm of the large ceramic tube should extend outside of the nut. Insert the lower Burner tube into the center of the large ceramic tube and finger tighten the heater swivel nut onto the tapered union fitting.

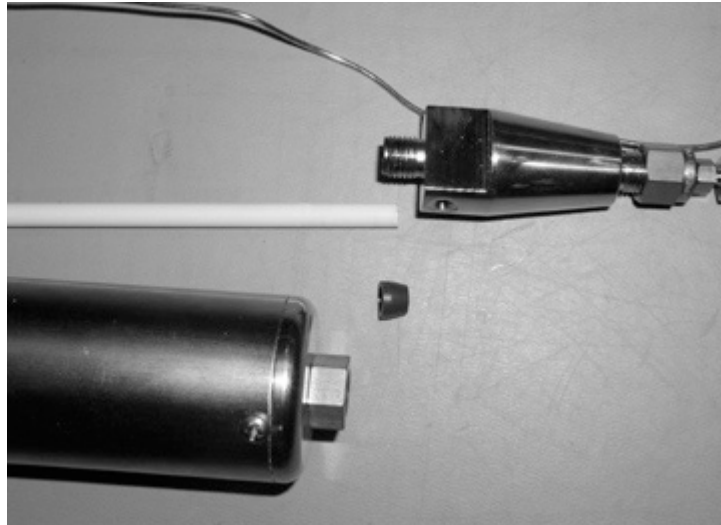


Figure 26 Proper Ferrule Orientation to the Large Ceramic Tube

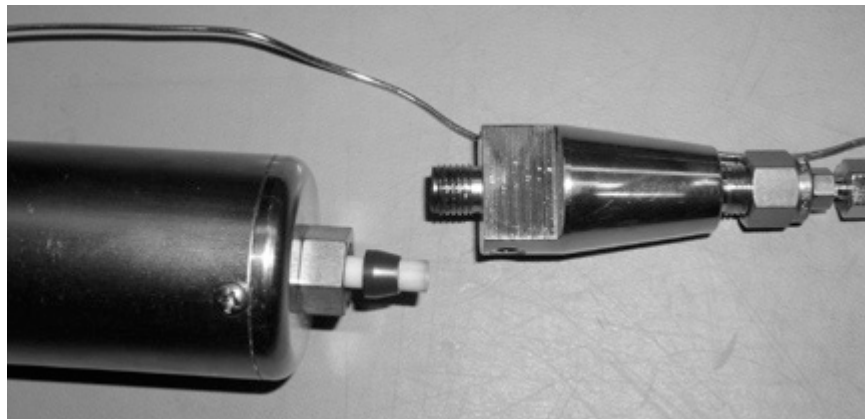


Figure 27 Large Ceramic Tube Properly Inserted into the Quartz Heater Assembly

- 7 Position the upper ceramic tube into the long axis of the splitter fitting so that about 4 mm of it extends past the top of the fitting. Slide the 0.054" ID double tapered ferrule over the upper ceramic tube. Gently holding these parts so that neither the ferrule nor the upper ceramic tube slips out of position, finger-tighten the union fitting onto the splitter fitting.

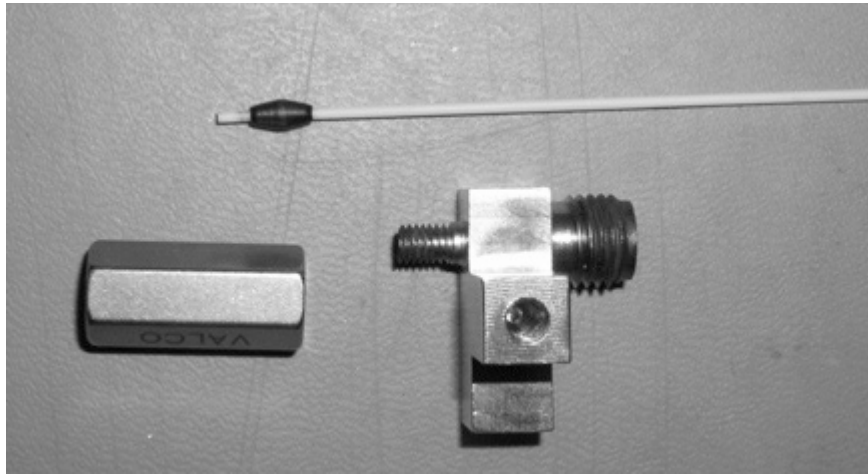


Figure 28 Orientation of the Double Taper Ferrule

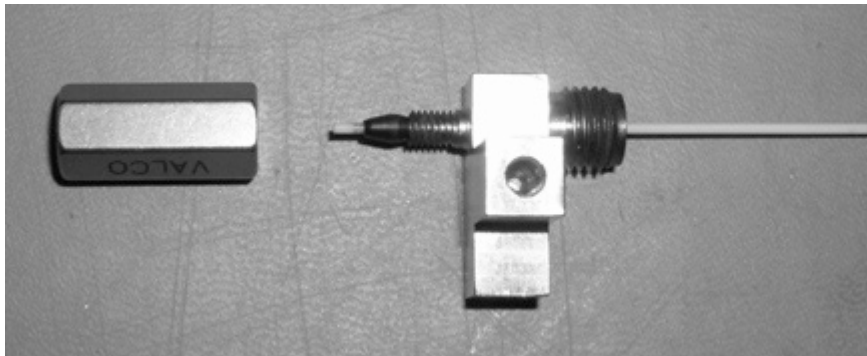


Figure 29 Positioning the Upper Tube in the Union Fitting

- 8** Approximately 1.5 cm of the large ceramic tube should extend above the top of the quartz heater assembly. Slide a 1/4" Swagelok nut over the large ceramic tube and then also slide a 1/4" ferrule over the tube (flat side on back of the nut).
- 9** Holding the splitter fitting, gently insert the upper ceramic tube into the large ceramic tube coaxially, to avoid placing stress on the fragile upper ceramic tube. Lower the splitter fitting into place to engage the threads of the 1/4" Swagelok nut. Tighten finger-tight.
- 10** To begin the final alignment and tightening, use a 7/16" wrench and 5/16" wrench to tighten the 1/4" Burner adapter one-quarter turn past finger-tight.
- 11** Using a 5/8" wrench on the heater swivel nut and a 1/2" wrench on one of the flats of the tapered union fitting, tighten the heater swivel nut one-quarter turn past finger-tight. Using a 5/16" wrench on the 1/4" Burner

adapter, rotate this fitting so that the brazed H₂ line is aligned 180 ° (opposite) from the oxidizer Inlet port.



Figure 30 Tightening the Heater Swivel Nut

- 12** Making sure that the Burner inlet fitting does not loosen, use a 1/2" wrench on a flat of the tapered union fitting and 9/16" wrench on the 1/4" Swagelok nut of the Burner adapter to tighten the tapered union fitting 1/4" past finger-tight.
- 13** Rotate the quartz heater assembly so that the thermocouple and heater leads are in the same plane and pointed in the same direction as the peg on the Burner inlet fitting. Turn the splitter fitting so that H₂ inlet port is also aligned with the peg on the Burner inlet fitting.

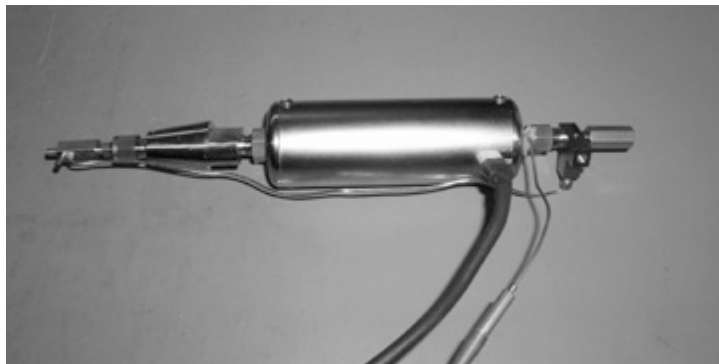


Figure 31 Proper Alignment of the Burner

- 14** Tighten the nut on the splitter fitting one-quarter turn past finger-tight using a 9/16" wrench on the 1/4" nut and a 7/16" wrench on the flats of the splitter fitting.

- 15** Carefully bend the H₂ line into position so that the 1/16" Valco nut and ferrule can be screwed into the side port of the splitter fitting. Tighten the connection of the H₂ line to the splitter fitting using a 3/8" wrench on the vertical flat of the splitter fitting and 1/4" wrench on the Valco nut.
- 16** Make sure that no other connections have loosened or moved out of alignment, if so, reposition or retighten the fittings as needed.
- 17** The assembled Burner is now ready for re-installation on the GC.

Assembling the Dual Plasma Burner for Component Replacement with the NCD

The following procedure can be used to assemble the Dual Plasma Burner for use with the NCD or for replacement of Burner components, such as ceramic tubes. Refer to Figure 2 on page 27 for proper part nomenclature. Note that the lower section of the NCD Burner is identical to the lower section of the SCD Burner.

NOTE

While the use of 1/4" ferrules is optional with the SCD, the quartz tube used in the NCD Burner is more fragile and the use of 1/4" graphite ferrules is highly recommended.

- 1 Slide the 0.066" internal diameter (I.D.) double taper ferrule onto the lower Burner tube. The tube should extend approximately 2 mm past the end of the ferrule.
- 2 Insert the lower Burner tube and double taper ferrule into the Burner inlet fitting.
- 3 Slide the 1/4" Burner adapter over the top of the lower Burner tube all the way down to the Burner inlet fitting and screw it onto the fitting finger-tight.

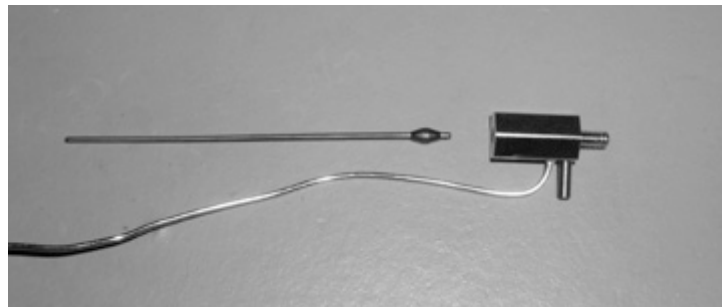


Figure 32 Ferrule Placement on Lower Burner Tube

- 4 Slide a 1/4" Swagelok nut over the Burner adapter and then slide a 1/4" ferrule over the Burner adapter and position it into the 1/4" nut.
- 5 Center the lower Burner tube so that it will slide into the tapered union. Insert the lower end of the tapered union fitting into the 1/4" Swagelok nut and screw it on finger-tight. If necessary, the brazed H₂ line can be gently bent out of the way. Be careful not to stress the brazed (welded) connection.
- 6 Find the quartz combustion tube, 1/4" ferrule and quartz heater assembly.
- 7 Insert the quartz tube into the quartz heater assembly. Position a 1/4" ferrule (flat end butted up against the top of the swivel nut) onto the quartz combustion tube. With the ferrule positioned against the swivel nut, approximately 0.5 cm of the quartz tube should extend outside the nut.

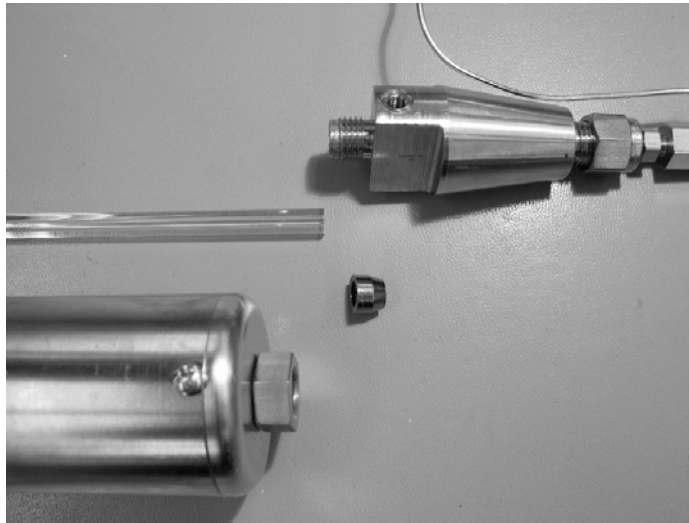


Figure 33 Proper Ferrule Orientation to the Large Quartz Tube

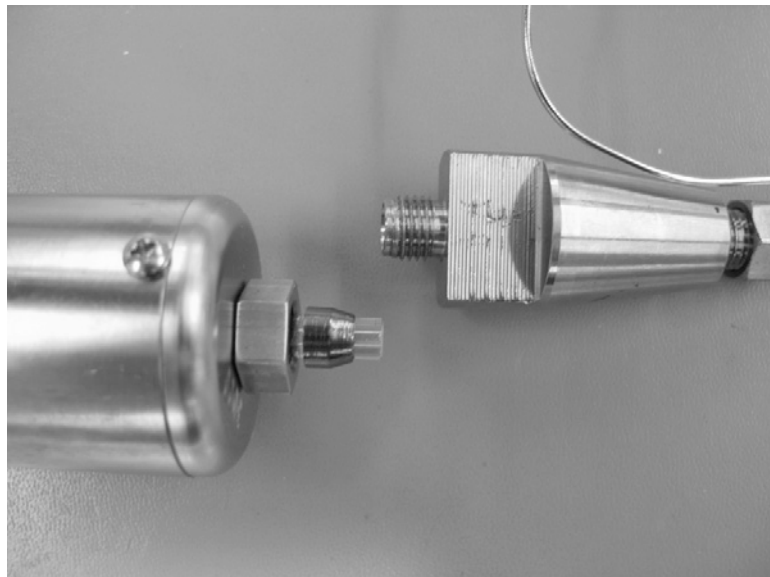


Figure 34 The Quartz Tube Properly Inserted into the Quartz Heater Assembly

- 8** Insert the lower burner tube into the center of the quartz tube and finger tighten the heater swivel nut onto the tapered union fitting, then tighten an additional 1/4 turn making sure not to break the quartz tube.
- 9** To begin the final tightening, use a 7/16" wrench and 5/16" wrench to tighten the 1/4" Burner adapter one-quarter turn past finger-tight.

- 10 Using a 5/8" wrench on the heater swivel nut and a 1/2" wrench on one of the flats of the tapered union fitting, tighten the heater swivel nut one-quarter turn past finger-tight. Using a 5/16" wrench on the 1/4" Burner adapter, rotate this fitting so that the brazed H₂ line is aligned 180 ° (opposite) from the oxidizer Inlet port.
- 11 Making sure that the Burner inlet fitting does not loosen, use a 1/2" wrench on a flat of the tapered union fitting and 9/16" wrench on the 1/4" Swagelok nut of the Burner adapter to tighten the tapered union fitting 1/4" past finger-tight.
- 12 Rotate the quartz heater assembly so that the thermocouple and heater leads are in the same plane and pointed in the same direction as the peg on the Burner inlet fitting. Turn the splitter fitting so that H₂ inlet port is also aligned with the peg on the Burner inlet fitting.

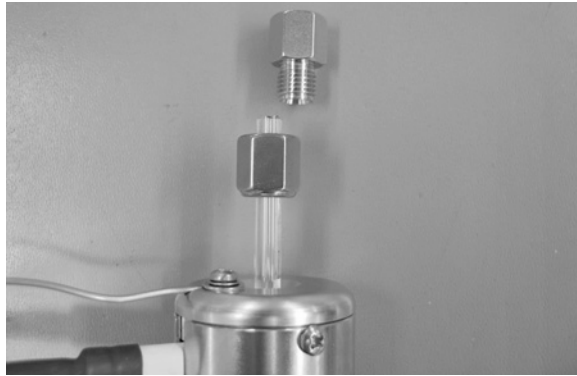


Figure 35 Burner Assembly Detail

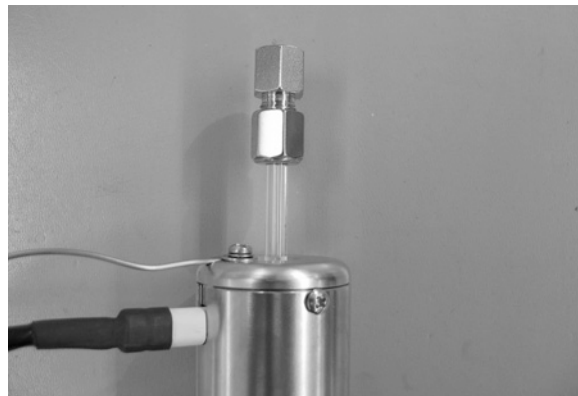


Figure 36 Burner Assembly Alignment

- 13 The assembled Burner is now ready for re-installation on the GC.

Tube Replacement for the SCD

The Burner has two combustion tubes that require replacement: the upper ceramic tube and the Large Ceramic Tube. Generally the tubes should be replaced only if sensitivity decreases. The “Troubleshooting” chapter provides additional information to assist in determining whether tube replacement may be necessary. Refer to Figure 1 on page 26 for proper part nomenclature. Follow the instructions below for tube replacement.

- 1 Turn off power to the GC and the Controller and let the system cool down under vacuum.
- 2 Turn off power to the vacuum pump.
- 3 Lift the Burner out of the shroud. It is recommended to remove the coil, noting the position of the ferrule. In some instances, if the coil can be uncoiled, it may be convenient to leave it attached to the Burner.
- 4 Disconnect the hydrogen and oxidant lines.
- 5 Disconnect the power connector that leads to the GC, if necessary.
- 6 Tilt the Burner at an angle, so that when loosening the union fitting the upper ceramic tube does not slide down into the large ceramic tube.
- 7 Loosen and disconnect the union fitting, and pull the splitter fitting and upper ceramic tube out of the Burner.
- 8 Slide the upper ceramic tube out of the splitter fitting.
- 9 Slide the upper ceramic tube into the splitter fitting, so that approximately 4 mm of the tube extends beyond the top of the fitting. Then, slide the double taper ferrule onto the tube (see Figure 37 and Figure 38 for proper positioning). Gently holding these parts so that neither the ferrule nor the upper ceramic tube slip out of position, finger-tighten the union fitting onto the splitter fitting.

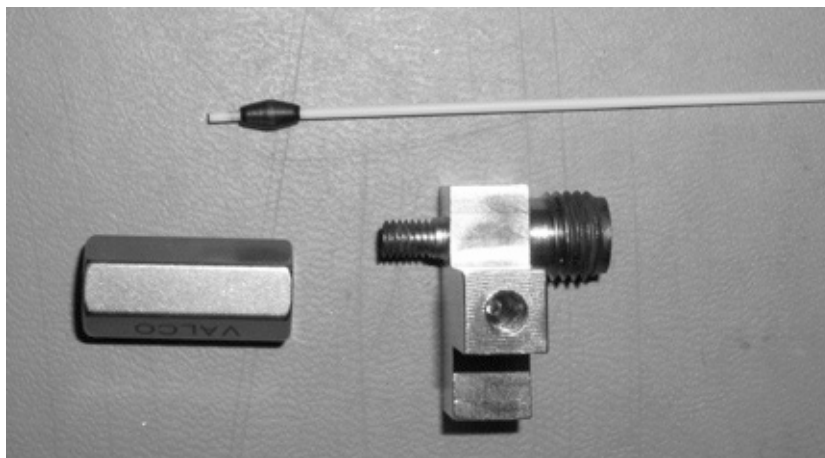


Figure 37 Orientation of the Double Taper Ferrule

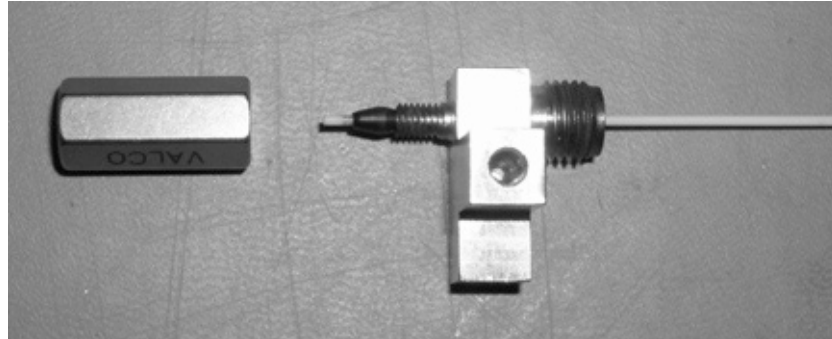


Figure 38 Positioning the Upper Tube in the Union Fitting

- 10** Holding the splitter fitting, gently insert the upper ceramic tube into the large ceramic tube coaxially, to avoid placing stress on the fragile upper ceramic tube. Lower the splitter fitting into place to engage the threads of the 1/4" Swagelok nut. Tighten finger-tight.
- 11** If you do not need to replace the Large Ceramic Tube, proceed to step 19.
- 12** Remove the Tapered Union Fitting from the bottom of the Burner.
- 13** Slide the Large Ceramic Tube out of the Burner and remove it from the Quartz Heater Assembly.
- 14** Insert the new large ceramic tube into the quartz heater assembly. Position a 1/4" ferrule (flat end butted up against the top of the swivel nut) onto the large ceramic tube. With the ferrule positioned against the swivel nut, approximately 0.5 cm of the large ceramic tube should extend outside of the nut. Insert the lower Burner tube into the center of the large ceramic tube and finger tighten the heater swivel nut onto the tapered union fitting.

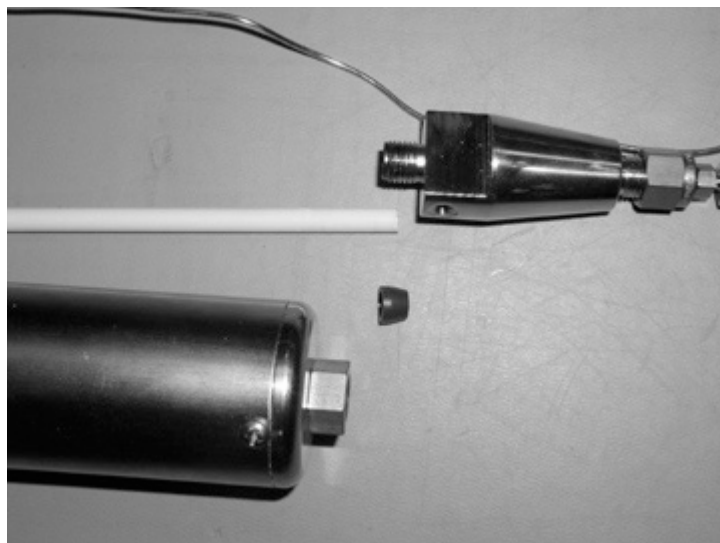


Figure 39 Proper Ferrule Orientation to the Large Ceramic Tube

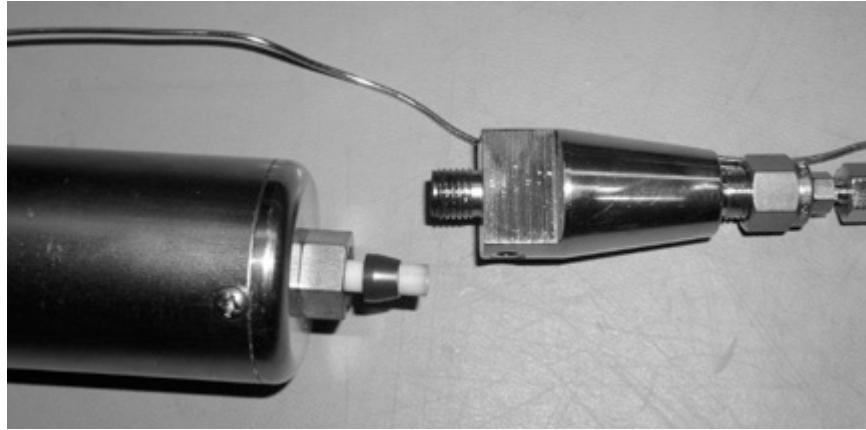


Figure 40 Large Ceramic Tube Properly Inserted into the Quartz Heater Assembly

- 15** Approximately 1.5 cm of the large ceramic tube should extend above the top of the quartz heater assembly. Slide a 1/4" Swagelok nut over the large ceramic tube and then also slide a 1/4" ferrule over the tube (flat side on back of the nut).
- 16** To begin the final alignment and tightening, use a 7/16" wrench and 5/16" wrench to tighten the 1/4" Burner adapter one-quarter turn past finger-tight.
- 17** Using a 5/8" wrench on the heater swivel nut and a 1/2" wrench on one of the flats of the tapered union fitting, tighten the heater swivel nut one-quarter turn past finger-tight. Using a 5/16" wrench on the 1/4" Burner adapter, rotate this fitting so that the brazed H₂ line is aligned 180 ° (opposite) from the oxidizer Inlet port.

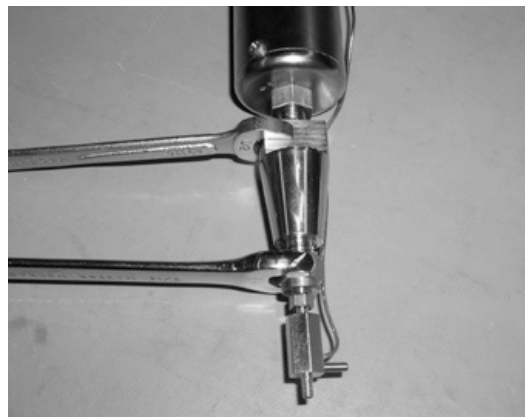


Figure 41 Tightening the Heater Swivel Nut

- 18** Making sure that the Burner inlet fitting does not loosen, use a 1/2" wrench on a flat of the tapered union fitting and 9/16" wrench on the 1/4" Swagelok

nut of the Burner adapter to tighten the tapered union fitting one-quarter turn past finger-tight.

- 19 Rotate the quartz heater assembly so that the thermocouple and heater leads are in the same plane and pointed in the same direction as the peg on the Burner inlet fitting. Turn the splitter fitting so that H₂ inlet port is also aligned with the peg on the Burner inlet fitting.



Figure 42 Proper Alignment of the Burner

- 20 Tighten the nut on the splitter fitting one-quarter turn past finger-tight using a 9/16" wrench on the 1/4" nut and a 7/16" wrench on the flats of the splitter fitting.
- 21 Carefully bend the H₂ line into position so that the 1/16" Valco nut and ferrule can be screwed into the side port of the splitter fitting. Tighten the connection of the H₂ line to the splitter fitting using a 3/8" wrench on the vertical flat of the splitter fitting and 1/4" wrench on the Valco nut.
- 22 Make sure that no connections have loosened or moved out of alignment, if so, reposition or retighten the fittings as needed.
- 23 Replace the Burner in the shroud.
- 24 Follow the standard system start-up procedure, including column placement.

Tube Replacement for the NCD

The Burner has one quartz combustion tube that requires replacement. Generally the tube should be replaced only if sensitivity decreases. The “Troubleshooting” chapter provides additional information to assist in determining whether tube replacement may be necessary. Refer to Figure 2 on page 46 for proper part nomenclature. Follow the instructions below for tube replacement.

- 1 Turn off power to the GC and the Controller and let the system cool down under vacuum.
- 2 Turn off power to the vacuum pump.
- 3 Lift the Burner out of the shroud. It is recommended to remove the coil, noting the position of the ferrule. In some instances, if the coil can be uncoiled, it may be convenient to leave it attached to the Burner.
- 4 Disconnect the hydrogen and oxidant lines.
- 5 Disconnect the power connector that leads to the GC, if necessary.
- 6 Loosen and disconnect the union fitting from the top of the Burner.
- 7 Remove the tapered union fitting from the bottom of the Burner.
- 8 Slide the large quartz tube out of the Burner and remove it from the quartz heater assembly.
- 9 Insert the new quartz tube into the quartz heater assembly. Position a 1/4" ferrule (flat end butted up against the top of the swivel nut) onto the quartz combustion tube. With the ferrule positioned against the swivel nut, approximately 0.5 cm of the quartz tube should extend outside the nut.

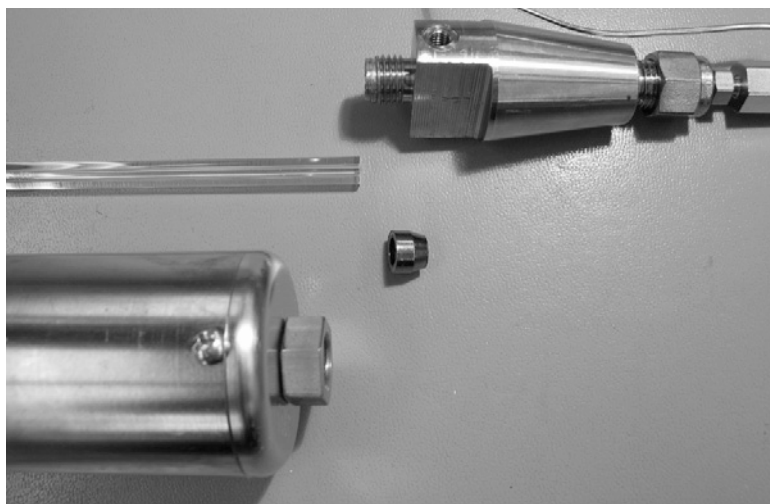


Figure 43 Proper Ferrule Orientation to the Large Quartz Tube

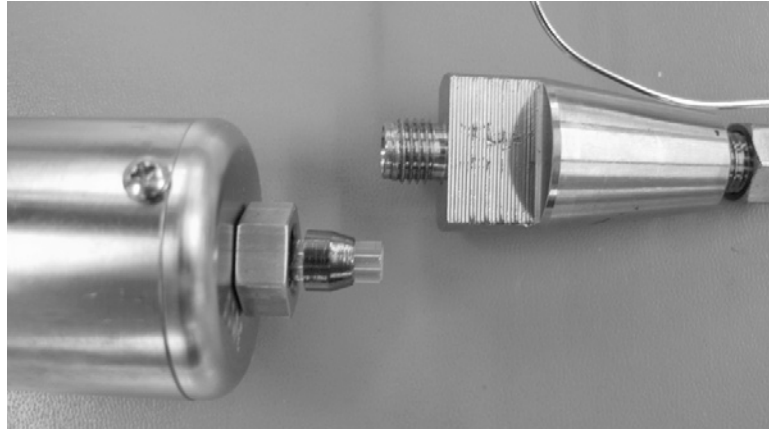


Figure 44 Large Quartz Tube Properly Inserted into the Quartz Heater Assembly

- 10** Insert the lower burner tube into the center of the quartz tube and finger tighten the heater swivel nut onto the tapered union fitting, then tighten an additional 1/4 turn making sure not to break the quartz tube.
- 11** To begin the final tightening, use a 7/16" wrench and 5/16" wrench to tighten the 1/4" Burner adapter one-quarter turn past finger-tight.
- 12** Using a 5/8" wrench on the heater swivel nut and a 1/2" wrench on one of the flats of the tapered union fitting, tighten the heater swivel nut one-quarter turn past finger-tight. Using a 5/16" wrench on the 1/4" Burner adapter, rotate this fitting so that the brazed H₂ line is aligned 180° (opposite) from the oxidizer Inlet port.
- 13** Making sure that the Burner inlet fitting does not loosen, use a 1/2" wrench on a flat of the tapered union fitting and 9/16" wrench on the 1/4" Swagelok nut of the Burner adapter to tighten the tapered union fitting one-quarter turn past finger-tight.
- 14** Rotate the quartz heater assembly so that the thermocouple and heater leads are in the same plane and pointed in the same direction as the peg on the Burner inlet fitting. Turn the splitter fitting so that H₂ inlet port is also aligned with the peg on the Burner inlet fitting.

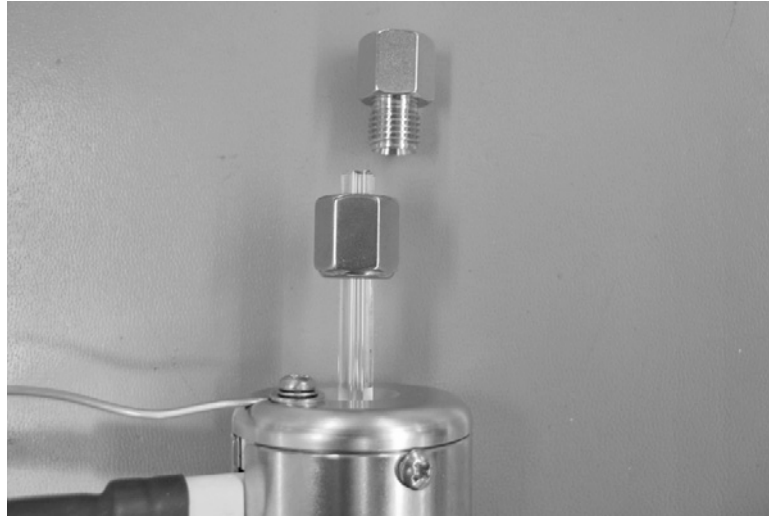


Figure 45 NCD Tube Replacement Detail

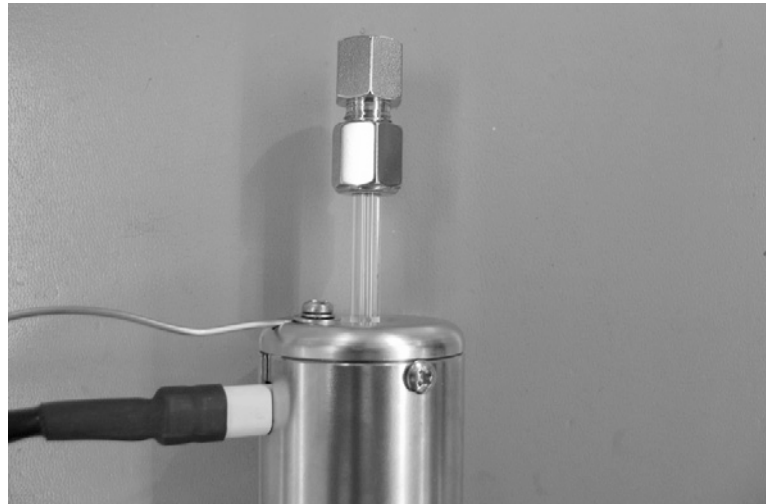
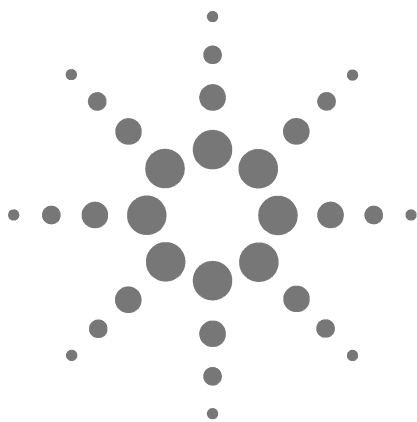


Figure 46 NCD Tube Replacement Detail

- 15** The assembled Burner is now ready for re-installation on the GC, including column placement.



7 Troubleshooting

Solving Detector Problems 106

Power Problems 107

Ozone Generation Problems 109

Response Problems 111

Temperature Reading Problems 112

Diagnosing General Problems 113



Solving Detector Problems

A basic understanding of the Detector helps one systematically diagnose and solve Detector problems. Many symptoms may be caused by more than one problem and these are the most difficult to troubleshoot. It should be pointed out, however, that analysis of sulfur or nitrogen compounds has traditionally been very difficult because of the inherent reactivity and instability of the compounds themselves. Often, problems blamed on the Detector actually originate from either poor chromatographic technique or other system failures (most of these problems are injector related).

Therefore, the first step in troubleshooting is to isolate the problem: in the chromatographic system, the Burner assembly, or in the Detector itself (ozone generator, vacuum pump, photomultiplier tube or electronics). Diagnosing the location of a problem is often facilitated by returning to Chapter 4 and attempting to verify initial conditions. The table at the end of this section lists many common problems, their most probable causes and corrective action that should be taken.

As a good practice, keep a maintenance log (as advised in Chapter 6) and use it to aid troubleshooting. In the maintenance log, keep track of detector flow rates (oxidizer and hydrogen), pressures (burner controller and reaction cell in detector), and background signal (the difference between ozone “on” and ozone “off”).

For assistance with troubleshooting Detector problems, contact Agilent with the serial number of the unit, the conditions used by the instrument, and any recent changes that have been made.

Power Problems

The first step in the determination of a power related problem is to verify power to the pump, Controller, and Detector itself. The inability to establish power may be as trivial as a blown fuse. Fuse requirements and positions on the Detector are indicated in see Figure 47 and Table 4.

Repeated fuse failure is an indication of a more serious situation.

Detector Fuse

If the Standby LED does not illuminate when the unit is plugged in, and multiple fuses fail on the Detector, disconnect the power cord and contact Agilent.

Vacuum Pump Fuse

Repeated blown fuses of the vacuum pump indicate a pump oil problem. If the vacuum pump has not been in operation for some time or if water accumulated in the vacuum pump has not been properly removed, the pump may be difficult to start, or may require more power than normal, and this may blow the main AC fuse. Disconnect the main power cord from the AC outlet and from the back panel of the Detector. Replace the pump oil. Connect the pump power cord to an AC outlet in the lab to start it. Allow the pump to operate for 10 to 15 minutes, after which the pump may be plugged back into the rear panel of the Detector. If fuses blow after the pump oil has been changed, the pump has been damaged. Contact Agilent for further information.

The dry piston pump may require a short break-in time when new or after seals have been changed. It can be operated for a few hours by plugging it into a wall outlet. When turned off, it is necessary to allow the pump pressure to reach atmospheric conditions before restarting.

Also note that the dry piston pump is equipped with a thermal fuse; connecting the pump to a power supply that exceeds 110 V \pm 10% will result in the pump shutting down.

Dual Plasma Controller Fuses

If the Controller will not power on, confirm that the power cord is firmly seated at both ends. Also make certain that there is power to the outlet at the wall, surge protector, or circuit breaker. If the power cord is seated correctly and power is available to the Controller, the fuse in the power entry module (just above the power switch) on the back of the Controller may have blown. Make sure that the actual line voltage and the setting on the Controller match.

To replace the fuse in the power entry module, follow these steps:

- 1 Unplug the power cord from the back of the Controller.
- 2 Use a small flat-head screwdriver to pry open the fuse cover on the power entry module on the back of the Controller.

3 Remove the old fuses from the holder and replace with new fuses.

If the Controller still does not power on after replacing the fuse, contact Agilent.

Consult Table 4 for the appropriate replacement fuse.

Table 4 Fuses for 100 V, 120 V and 230 V Versions of 355 SCD and 255 NCD

	100/120 V	230 V
Detector Back Panel		
Main AC Power	15 A/250 V 3AG slo-blo	T10 A/250 V 5 x 20mm
Detector		
Electronics Power Supply (F1)	250 mA/250 V 3AG slo-blo	T125 mA/250 V 5 x 20mm
Pump (F2)	15 A/250 V 3AG slo-blo	T5 A/250 V 5 x 20mm
Ozone Generator (F3)	1 A/250 V 3AG slo-blo	T100 mA/250 V 5 x 20mm
Photomultiplier Tube Cooler	1 A/250V 3AG slo-blo	T500 mA/250V 5 x 20mm
Dual Plasma Controller Back Panel		
Main AC Power (2 fuses)	T2 A/250 V 5 x 20mm	1 A/250 V 5 x 20mm

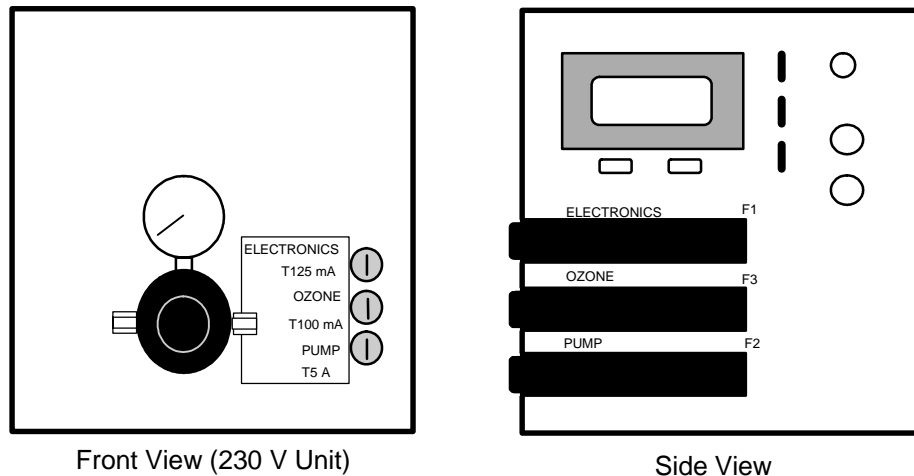


Figure 47 Fuse Positions on the Power Supply Board

Ozone Generation Problems

Following the verification of power to the instrumental components, the next step in tracing a problem is the determination of ozone production. With the ozone toggle off and the vacuum pump on, read the signal output on the front of the Detector. Toggle the ozone on. A properly operating Detector will typically display a difference in background of 0.2 to 2 mV. If no change is observed, a problem most likely lies within the ozone generator itself, the high voltage transformer supplying power to the ozone generator, or in the transfer line system between the ozone generator and the reaction cell.

Ozone Generator

Loss of ozone from the ozone generator may arise from generator fault, or from a leak in the corona discharge tube. Typically a pungent ozone odor will be noticed emanating from the side of the Detector if the ozone generator is leaking.

WARNING

Any operation requiring access to the inside of the equipment, could result in injury. To avoid potentially dangerous shock, disconnect from power supply before opening the equipment.

WARNING

High voltages supply the ozone generator. Unplug the instrument.

Removal of the left side panel (as seen from the front of the Detector) and removal of the ozone generator cover panel may reveal corrosion caused by escaped ozone. If this is the case, replace the ozone generator. Resistance across the leads to the ozone generator should be infinite. Measure the resistance with an ohmmeter to verify. If there is a resistance, replace the ozone generator.

High Voltage Transformer

WARNING

The high voltage transformer produces 6000 - 8000 volts.

The following test should only be performed by those with high voltage experience using high voltage probes.

Using high voltage probes, directly measure the secondary output of the ozone generator. A properly functioning generator will produce approximately 7500 volts. Output less than 3000 volts mandates replacement of the high voltage transformer.

Plugged Restrictor Lines

Plugged restrictor lines are verified by turning off the air/oxygen regulator located inside the door of the Detector and observing little or no change in the pressure as indicated on the regulator. The plug can be located in either the pre-ozone restrictor or the post-ozone restrictor (see Figure 3). A partially plugged restrictor line can result in a non-linear signal, or reduced sensitivity which will vary with the flow of ozone. Replace the restrictor lines.

Response Problems

Low or no response problems are the most difficult to troubleshoot on the Agilent SCD and NCD as they may arise from one source or a combination of sources. Primarily, response problems are due to combustion problems, and Burner integrity should be investigated first.

It is beyond the scope of this supplement to deal with all possible chromatography related problems. The following table lists many of the common problems, their most probable causes, and the corrective action that should be taken. For further assistance contact Agilent.

Temperature Reading Problems

Normally, the controller displays the burner temperature set-point. For diagnostic purposes, a switch in the controller (JP2) can be positioned to allow the display of the actual thermocouple temperature reading. When the actual temperature and set-point temperature agree, the heater indicator light on the front of the controller is illuminated. Note that there is a slight offset between these readings.

Diagnosing General Problems

Table 5 Troubleshooting Detector Issues

Problem	Possible Cause	Diagnosis	Corrective Action
No Response	No ozone	Little or no difference in output signal between ozone ON vs. OFF. Typically the background signal will be 0.3-0.8 mV higher with the ozone ON.	Continue below. See No Ozone in Problem column.
No Ozone	Blown fuse	Ozone indicator light remains off when ozone button is pushed.	Replace ozone fuse.
No Ozone	High Voltage Transformer and/or ozone generator is inoperative.	No difference in output signal between ozone ON vs. OFF even though flow through the ozone generator is good (about 25-35 mL/min).	Have ONLY someone with high voltage experience use a high voltage probe to check the input to the ozone generator across the two wires. It should be >6000 volts. If it is not then replace high voltage transformer. If it is, replace ozone generator.
No Ozone	Ozone restrictor(s) plugged.	Needle on the regulator inside front panel does not move downward when regulator is turned fully counterclockwise. The measured flow is low or non-existent.	Replace plugged restrictor(s).
No Response	Hydrogen and/or air ran out.	Measure flow rates.	Set correctly.
No Response	Broken ceramic tube(s).	Pressure on Controller and/or Detector is too high or too low.	Change tubes.
No Response	Heating element failure.	Temperature at Controller <100 °C.	Replace heating element.
Low Response	Improper hydrogen/air flow rates.	Measure flow rates.	Adjust flow rates.
Low Response	Detector leaks.	Note pressures on Controller.	Locate and repair leaks, check integrity of ferrules.

Table 5 Troubleshooting Detector Issues (continued)

Problem	Possible Cause	Diagnosis	Corrective Action
Low Response	Contaminated ceramic tubes.	If there does not appear to be a leak, then the tubes should be inspected. Contamination can result from column bleed, samples which may contain volatile metal complexes, and large injections of coke forming hydrocarbons.	Replace tubes.
Wandering Baseline	Poor temperature control.	Monitor the temperature on the Controller. It should vary by no more than ± 5 °C.	Check for a loose connection on the heating element or thermocouple. Reposition the thermocouple in the Burner.
Wandering Baseline	Contamination in one of the Detector gases.	Check the difference in the output signal between ozone on and off. The difference should be between 0.2-2 mV after equilibration.	Change Detector gases after adding in-line traps.
Wandering Baseline	Leak in the oxidizer line at the oxidizer inlet connection. Leak at weld of H ₂ inlet fitting.	If using SCD, use a microliter syringe containing a small amount of sulfur compound e.g., CS ₂ to "snoop" for leaks while watching mV output. Leaks are very evident by a large increase in signal displayed on the LED.	Reseat air line into air inlet nut, or replace.
Tailing Peaks with Non-Equimolar Response	Severe contamination of Detector gases.	High background signal with respect to ozone off.	Avoid "house" gases, especially air from compressors. Clean gases should be used with appropriate traps.
Tailing Peaks	Poor column connection.	Verify column position at inlet and outlet. Look for discoloration of column at Detector side which indicates column in combustion zone.	Reinstall column.
Tailing Peaks	Cracked tubes.	Confirm pressure and vacuum ranges. Inspect columns and ferrules.	Replace tubes as needed.
Controller Reads >1000 °C	Thermocouple open.	Check electrical resistance between thermocouple pins.	Replace thermocouple.

Table 5 Troubleshooting Detector Issues (continued)

Problem	Possible Cause	Diagnosis	Corrective Action
Burner Fault Cycles. (off and ON)	Cracked tube.	Pressure increases >525 torr, Burner faults. Cycle repeated as temperature and pressure reset.	Replace broken tubes.
Burner Fault Cycles (off and ON)	Leak in Burner.	Pressure increases >600 torr, Burner faults. Cycle repeated as temperature and pressure reset.	Locate and repair leaks, check integrity of ferrules.

Table 6 Troubleshooting Pump Issues

Problem	Possible Cause	Diagnosis	Corrective Action
Pump is noisy	High-pressure dry piston.	Pressure in the reaction cell is unacceptably high and the pump is getting audibly noisy.	Change sleeve/seal.
Pump Doesn't Start	Dry piston seals have recently been changed.	Pump fuses blow immediately after changing the seals and sleeves.	Dry piston requires break-in time of a few minutes after seals are changed.
Pump Doesn't Start	Pump switch off (dry piston pump must start from atmospheric pressure).	Locate pump switch on pump and verify position.	Turn ON switch.
Fuses Blow on Startup	Emulsified oil (worn seals and head on dry piston pump).	Inspect oil for integrity.	Change pump oil, and plug unit into wall to run for 10-15 minutes.
Water in Pump	Cracked coalescing filter.	Milky yellow oil in the pump window.	Change coalescing filter and pump oil.
Reaction Cell Pressure High	chemical trap clogged.	Remove trap from the vacuum line and confirm expected pressure readings.	Change chemical trap.
Reaction Cell Pressure High	Leak in ozone generator.	Air regulator falls rapidly to zero when turned off.	Replace ozone generator.
Pump Loses Oil Gurgle Sound	Ballast Open.	Oil level drops.	Reset ballast See Pump specific sections.
High level of oil in Coalescing Filter	Plugged oil return restrictor	No visible movement of oil in the return line.	Change filter and clear restrictor.

Table 7 Troubleshooting Burner Issues

Problem	Possible Cause	Diagnosis	Corrective Action
Fuses Blow on Startup	Shorted heater element.	Inspect for exposed wires or broken insulation around wire leads. Measure resistance leads, should not be 0.	Take Burner top off and reseal insulation around heater leads.
Low Sensitivity	Leaks at ferrules or fittings.	For SCD, use a microliter syringe containing a small amount of sulfur compound e.g., CS ₂ to "snoop" for leaks while watching mV output. Leaks are very evident by a large increase in signal displayed on the LED. For NCD, a high background could be observed from atmospheric nitrogen.	Inspect fittings for scoring and replace if necessary. Replace ferrules if worn. Hand tighten all fittings mounted onto vespel ferrules, and wrench tighten all connections with Valco ferrules.
Low Sensitivity	Improper gas flows.	Measure gas flows.	Reset gas flows.
Low Sensitivity High Pressure	Broken ceramic tubes or ferrules	Remove and inspect tubes and ferrules for chips and/or breaks.	Replace tubes or ferrules.
Low Sensitivity High Background	Improper column positioning.	Inspect for charred Detector end of column.	After cutting off charred end of column, measure column length 108-109 mm from ferrule (or 114-115 mm from bottom of nut) and re-attach column.

Index

C

Calibration of Flow Sensor, 88
Ceramic Tube Replacement, 98
Column Insertion Distance, 61

D

Detection Limits, 76
Detector
 cleaning, 83
 display output controls, 65
 initial check, 69
 operating conditions, 75
 operation, 73
 power controls, 64
 schematic, NCD, 36
 schematic, NCD (nitrosamine mode), 37
 schematic, SCD, 35
 shut down, 77
 signal controls, 65
 start-up procedure, 72
Dry Piston Pump
 installation, 52
Dual Plasma Burner
 assembling for NCD, 95
 assembling for SCD, 90
 cross-section illustration for NCD, 27
 cross-section illustration for SCD, 26
 installing, 59
 overview, 25
 tube replacement for NCD, 102
 tube replacement for SCD, 98
Dual Plasma Controller
 controls and settings, 66
 front panel illustration, 66
 installing, 60
 operating conditions, 75
 overview, 24
 rear panel illustration, 60

E

Edwards oil-sealed pump
 ballast position, 51
 installation, 46

F

FID Adapter
 overview, 33
Flow Sensor Calibration, 88

Fluidics overview, 42
Fuses
 replacing, 107, 108

I

Installation
 column connections, 61
 Dry piston pump, 52
 Dual Plasma Burner, 59
 Dual Plasma Controller, 60
 Edwards oil-sealed pump, 46
 gases, 42, 43
 initial connections, 46
 overview, 40
 oxidant supply, 57
 power requirements, 41
 required tools, 45
 selecting a location, 41
 signal output cables, 58
 transfer line, 62
 Vacuum Pump, 46
 Welch dry piston pump, 52

M

Maintenance
 changing oil mist filter, 84
 cleaning the detector, 83
 pump, 82
Minimum Detection Limit (MDL), 89

O

Operating Conditions, 75
Ozone Generator
 overview, 28
 troubleshooting, 109

P

Pressure Regulator, use of, 43

Q

Quartz Tube Replacement, 102

R

Recorder test, 68

S

Schematic
 for NCD, 36
 for NCD (nitrosamine mode), 37
 for SCD, 35
Sensitivity
 minimum detection limit, 89
 NCD specifications, 20
 SCD specifications, 20
Sensor Calibration, 88
Shut-down procedure, 77
Signal Output Cables
 installation, 58
Special Operating Modes
 nitrosamine, 78
 nonhydrocarbon gaseous samples, 78
Specifications, 20
Start-up Procedure, 72
System schematic, 42
System Stabilization, 73
System Startup, initial, 67

T

Temperature
 set-point vs. thermocouple reading, 112
Theory of Operation, 23
Troubleshooting
 burner issues, 116
 detector, 106
 detector issues, 113
 general problems, 113
 ozone, 109
 power and fuses, 107, 108
 pump issues, 115
 table of problems and causes, 113
Tube Replacement
 for NCD, 102
 for SCD, 98

V

Vacuum Pump
 overview, 32

W

Welch Dry Piston Pump
 installation, 52
 operation notes, 53



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